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TEXTILE RESEARCH JOURNAL

VOL. XVIII, No. 9 September, 1948

The Visco-Elastic Properties of Wool Fibers*

L. Peters and J. B. Speakman

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A SATISFACTORY INTERPRETATION of the long-range elasticity of rubber is provided by the kinetic theory of elasticity. The chain molecules, which are in various states of contraction due to thermal agitation, are straightened when tension is applied, and the decrease in entropy accounts for the increase in load at high extensions. Elaboration of the theory is unnecessary because the work of Kuhn [7], Wall [13], and Treloar [12] is well known, but attention should be drawn to the fact that it has been used by Guth and James [3] to derive the following expression for the tension in stretched rubber:

$$f = C\left(\alpha - \frac{1}{\alpha^2}\right), \qquad (1)$$

where f is the tension, α is the degree of elongation, and C is a constant. A more complicated equation, describing a sigmoid stress-strain curve, has recently been obtained by Huggins [6].

As has been shown by Bull [2] and Woods [15], the statistical theory of rubberlike elasticity cannot be applied to keratin, because, in this instance, entropy plays only a small part in elastic recovery. In addition, the theory fails to explain viscous effects such as gradual extension under constant stress, delayed elasticity, and decay of tension at constant extension, all of which are especially pronounced in the case of wool. To explain such phenomena, Tobolsky and Eyring [11] pictured the long-chain molecules of rubber as being cross-linked by bonds of fairly low energy, which are continually being broken and re-formed under the influence of thermal energy. Applied tension increases the rate of bond breakdown and the probability of re-formation on new sites, thus causing plastic flow. Molecules which are prevented from slipping by high intermolecular attraction act as simple springs according to the kinetic theory of elasticity, while molecules which both stretch and slip act as Maxwell visco-elastic elements. Assuming that these two kinds of units act in parallel, as shown by the three-element model of Figure 1, a successful interpretation of the behavior of rubber was obtained. The theory was therefore extended to wool and other textile fibers by Eyring, Halsey, and coworkers [5]. In order to describe the precise behavior of wool, however, Halsey and Eyring [4] found it necessary, in a later paper, to postulate arbitrary non-Hookean springs and a more com-

* This paper was read at a meeting of the British Rheologists' Club on March 20, 1948.

plicated model than that of Figure 1. The absence of a satisfactory physical counterpart of so complex an analogy suggested that this type of approach to the problem of interpreting the properties of wool should be abandoned in favor of one based on its known molecular structure.

The long peptide chains of wool are linked by cystine and salt linkages to form grids which, according to Astbury and Bell [1], are folded as shown in Figure 2.

Stability is given to the fold by cross-linkages between the component chains of the grid, by attractive forces between neighboring side-chains, and by hydrogen bonds within the fold. Cohesion between the folded grids is provided by hydrogen bonds between neighboring peptide groups and van der Waals forces. Although the structure shown in Figure 2 was deduced from x-ray observations, it is customary to assume that the folded grid is a feature of both the amorphous and the crystalline regions of the wool fiber, which then differ merely in degree of perfection of arrangement of the grids. When the fiber is stretched, extension is confined in its early stages to the less-ordered regions, but at extensions above about 20 per cent the crystalline phase is gradually transformed from the folded (α-) into the straight (β-) configuration. As the process of unfolding occurs against a variety of cohesive forces within and between grids, it is obvious that the elastic properties of wool will be extremely complex—so complex, in fact, as to suggest that a quantitative interpretation of behavior in terms of the known molecular structure is unlikely to be realized.

Fortunately, the problem can be simplified by confining attention, in the first instance, to the elastic properties of wool fibers in concentrated solutions of weak acids. The swelling pressure developed in such media is great enough to separate the grids from one another. In 98-percent formic acid, for example, wool fibers increase in diameter by about 50 percent, with only a slight change in length [8], and the separation of the grids is established by the failure of the swollen fibers to give an x-ray fiber diagram. Hence the elastic properties of wool fibers in formic acid are, in effect, those of isolated grids. Once their properties have been determined, the effect of superimposing grids can be isolated by comparing the behavior of fibers in formic acid and water, or, better, in formic acid and, say, hydrochloric acid at the same pH value. The following interpretation of the elastic properties of wool was obtained in this way.

General Theory

It seems reasonable to assume that in the main peptide chains or grids of unstretched wool there is an equilibrium between α-folds (Figure 2) and occasional open folds of the form of β-keratin. If each α-fold is held in that state by bonds of some kind—for example, hydrogen bonds—there will tend to be a disproportionate number of segments or chains in the α-form. Writing ΔG_1 for the activation energy of the unfolding process and ΔG_2 for that of refolding, and assuming that the rate of each process is proportional to the number of folds to be transformed, the rate of unfolding at any time t is given by the expression:

$$\frac{d\beta}{dt} = \frac{kT}{h}(1 - \beta)e^{-\Delta G_1/RT} - \frac{kT}{h}(\beta)e^{-\Delta G_2/RT}, \quad (2)$$

from the theory of absolute reaction rates, where β is the fraction of the chains in the unfolded form. Integration of this equation leads to unwieldy solutions when the activation energies are affected by a constant rate of increase of stress. Simpler equations are obtained when the stress is constant, but the analysis is, as yet, incomplete and only

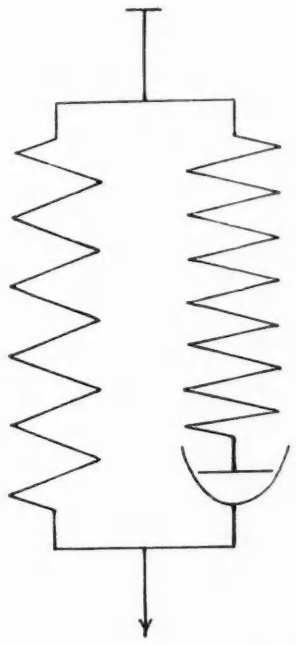

FIG. 1. *Tobolsky-Eyring-Halsey three-element model.*

general conclusions will be outlined at the end of this paper. All such difficulties can be avoided, however, by dealing with equilibrium conditions, for when $\frac{d\beta}{dt} = 0$, equation (2) simplifies to*

$$\frac{1 - \beta}{\beta} = e^{\Delta G/RT}, \tag{3}$$

where $\Delta G = \Delta G_1 - \Delta G_2$ (see Figure 3) and is the Gibbs free energy change per mole for the process of unfolding.

If E' is the equilibrium extension, expressed as a fraction of the length of the fiber when the chains are completely folded (pure α-keratin), and E_∞ is the extension when all the chains are completely transformed into β-keratin,

$$\beta = \frac{E'}{E_\infty}. \tag{4}$$

On inserting this relationship into equation (3), the following expression is obtained:

$$E' = \tfrac{1}{2}E_\infty \left(1 - \tanh \frac{\Delta G}{2RT} \right). \tag{5}$$

When there is no load on the fiber, $\Delta G = \Delta G^0$ and there will remain a residual extension E_0 such that:

$$E_0 = \tfrac{1}{2}E_\infty \left(1 - \tanh \frac{\Delta G^0}{2RT} \right). \tag{6}$$

Extensions are usually measured from the length under zero tension, and if E represents the extension from such an origin,

$$E = \tfrac{1}{2}E_\infty \left(\tanh \frac{\Delta G^0}{2RT} - \tanh \frac{\Delta G}{2RT} \right). \tag{7}$$

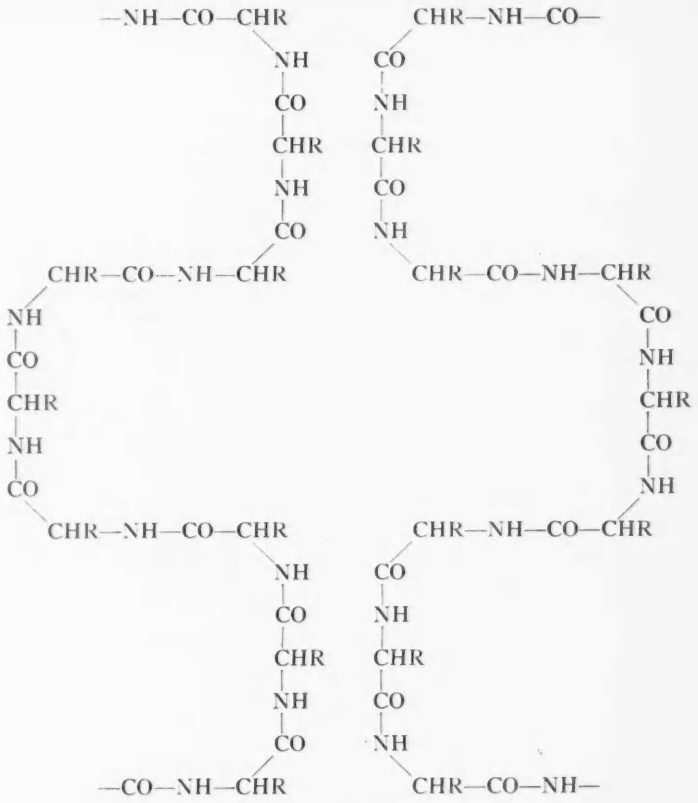

FIG. 2

When tension is applied to the ends of a chain, each fold will gain energy by moving in the direction of the force—that is, by unfolding—and lose energy by refolding. If the points in the fold which were held together by a bond of some kind move apart a distance 2λ, the gain in energy is $2\lambda\lambda'\lambda''f$, where $\lambda'\lambda''$ is the area of cross section of a single chain and f is the tension expressed as the force per unit area of cross section of the fiber. Hence, if the energy barrier is deformed symmetrically (Figure 3),

$$\Delta G_1 = \Delta G_1{}^0 - \lambda\lambda'\lambda''Nf$$

and (8)

$$\Delta G_2 = \Delta G_2{}^0 + \lambda\lambda'\lambda''Nf,$$

where N is Avogadro's number, since ΔG_1, ΔG_2, etc. are changes in molar free energy. Hence,

$$\Delta G = \Delta G^0 - 2\lambda\lambda'\lambda''Nf \tag{9}$$

and equation (7) may be written:

$$E = \tfrac{1}{2}E_\infty \left[\tanh \left(\frac{\lambda\lambda'\lambda''}{kT} f - \frac{\Delta G^0}{2RT} \right) + \tanh \frac{\Delta G^0}{2RT} \right]. \tag{10}$$

* AUTHORS' NOTE: Prior to the reading of this paper at a meeting of the British Rheologists' Club on March 20, Burte and Halsey had already derived equations (2), (3), and (4) in a paper which appeared in the September, 1947, issue of TEXTILE RESEARCH JOURNAL, page 465. Owing to the late arrival of journals from the United States, Burte and Halsey's paper was not seen by us until after our paper had been read, and it is regretted that their work received no acknowledgment. As would be expected, since our paper is based on the earlier work of Eyring and Halsey, there are many close similarities between this section of our paper and Burte and Halsey's prior publication, although the equations have been used for very different purposes, as will be clear from what follows. However, it should be mentioned that Burte and Halsey's paper meets many of the objections to the earlier papers of Halsey and Eyring which we had in mind when the present investigation was undertaken.

TABLE I

Time (min.) t	Load ($g.\times10^5$ per cm^2 initial area) f	Extension (%) E	E_1	E_2-2	$\gamma=1-\frac{E_2}{50}$	$\tanh^{-1}\gamma$
0.5	0.62	9.1	8.14	0.96	0.9408	1.745
1.0	1.24	16.4	15.29	1.11	0.9258	1.628
1.5	1.85	21.4	20.83	0.57	0.9486	1.818
2.0	2.47	25.2	24.86	0.34	0.9532	1.866
2.5	3.09	27.9	27.56	0.34	0.9532	1.866
3.0	3.71	30.6	29.28	1.32	0.9336	1.686
3.5	4.33	32.4	30.36	2.04	0.9192	1.584
4.0	4.95	34.1	31.01	3.09	0.8982	1.463
4.5	5.56	36.2	31.41	4.79	0.8742	1.351
5.0	6.18	37.9	31.65	6.25	0.8350	1.205
5.5	6.80	39.8	31.79	8.01	0.7998	1.098
6.0	7.42	42.0	31.87	10.13	0.7574	0.990
6.5	8.04	44.3	31.93	12.37	0.7124	0.892
7.0	8.66	47.8	31.95	15.85	0.6430	0.763
7.5	9.27	51.4	31.97	19.53	0.5694	0.647
8.0	9.89	55.5	31.99	23.51	0.4900	0.536
8.5	10.50	60.3	32.00	28.3	0.3940	0.417
9.0	11.12	65.2	32.00	33.2	0.2960	0.305

The form of the stress-strain curves for different values of ΔG^0 is shown in Figure 4. When ΔG^0 is large, the characteristic sigmoid shape of the usual load-extension curve is obtained, but it must be emphasized that the experimental curves normally include the effects of creep which receive no consideration in this simple equilibrium theory. When $\Delta G^0 = 0$, however, the Hooke's law region of the load-extension curve is missing and there is merely a simple unfolding from a 50 percent extended form (curve C). In this case, equation (10) takes the simple form:

$$E = \tfrac{1}{2}E_\infty \tanh \frac{\lambda\lambda'\lambda''}{kT} f. \qquad (11)$$

The application of these equations to the load-extension curves of wool fibers in formic acid and saturated air will now be examined.

Behavior of Wool Fibers in Formic Acid

As has already been mentioned, the swelling of wool fibers in formic acid is great enough to separate the folded grids from one another. Although it can, therefore, be expected that the behavior of the fibers in formic acid will be far simpler than in water, the fact that the main peptide chains are bridged by cystine linkages must be taken into account when the above equations, which were derived for free chains, are applied to the load-extension curves of wool fibers in formic acid. Those parts of the grid where disulfide bonds are infrequent will tend to behave as groups of independent chains and show less resistance to unfolding than regions which are heavily cross-linked. The equations can, however, be applied to both regions because the effect of cross-linking is merely to increase the mean energy required for unfolding and to reduce the distance moved during the process.

When a wool fiber was stretched by means of the fiber balance in 98-percent formic acid at 22.2° C, the data given in the first three columns of Table I were obtained [8].

As shown in Figure 5, the early form of the load-extension curve indicates that $\Delta G^0 \sim 0$ and that $\beta \sim \frac{1}{2}$ even under zero tension. If this section of the curve may be attributed to the less heavily cross-linked regions of the grids, as seems reasonable, deviation at higher loads from the simple behavior represented by curve C of Figure 4 will be due to unfolding of the heavily cross-linked regions as their higher energy barrier is overcome by the greater force. The point of inflexion on the load-extension curve of Figure 5 indicates an extension of about 32 percent for the first phase. In order to derive the load-extension curves of the two regions, an approximate value for $\lambda\lambda'\lambda''/kT$ was first chosen to fit equation (11) to the early part of the load-extension curve. The calculated values of the extension under various loads were then subtracted from the actual extensions, and the

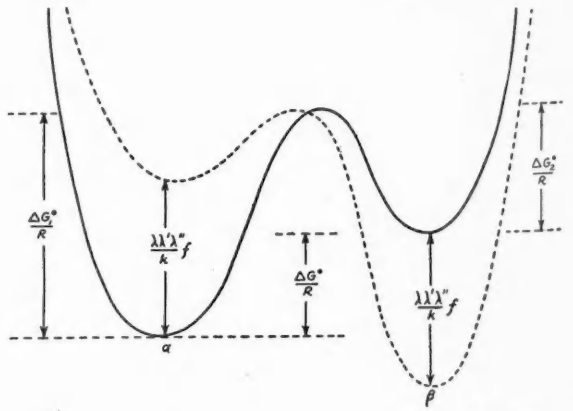

FIG. 3. *Free energy changes from folded to unfolded state.*
——— *initial state*
- - - - - - *stressed state*

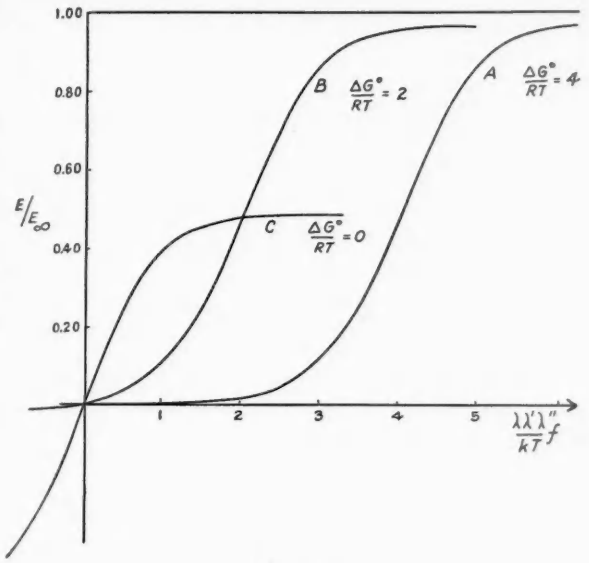

FIG. 4.

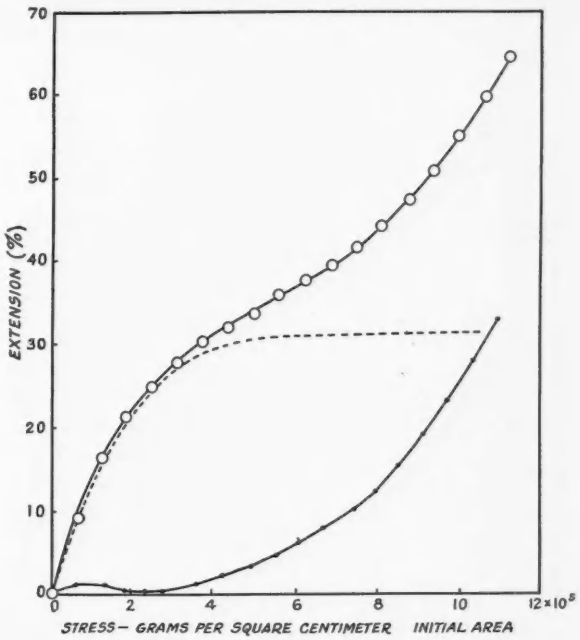

FIG. 5. *Extension of wool in concentrated formic acid at 22.2° C.*

difference curve had a form similar to that of the lowest curve in Figure 5. This resembles the first part of the high-energy curve A in Figure 4, but only an approximate value for the maximum possible elongation can be obtained. Using this value, however, better values for the extension of the first phase under different loads were obtained. The data, which are given in the column headed E_1 in Table I, are satisfactorily described by the following equation:

$$E_1 = 32 \tanh (0.42f), \qquad (12)$$

where f is expressed in g. $\times 10^5$ per square centimeter initial area. From equation (11), therefore, it is obvious that this phase should be capable of causing 32 percent contraction of the fiber when complete folding is induced. The reversible 30 percent supercontraction of wool fibers in cuprammonium solution [14] may well be of this type, and, as regards extension, it seems significant that the elastic properties of the fibers undergo no permanent alteration unless the extension exceeds about 30 percent with a high rate of loading [10].

Using Astbury's values for λ' and λ'', the distance moved by each point of an unfolding unit during the extension of the first phase may be calculated from equations (11) and (12):

$$\lambda = \frac{0.42 \times 1.371 \times 10^{-16} \times 295.2}{981 \times 10^5 \times 9.8 \times 10^{-8} \times 9.3 \times 10^{-5}} \text{ cms.}$$
$$= 1.9 \text{ Å.}$$

Since the value of λ is so small, it seems probable that the unfolded chains have the form of β-keratin. This, in turn, implies that β-keratin is present in unstretched fibers, because, according to the present theory, the phase under consideration is 50 percent unfolded in the absence of stress. X-ray examination of unstretched wool fibers in air, however, fails to reveal the presence of β-keratin, and it might on this account be supposed that the high swelling of wool fibers in formic acid is itself responsible for unfolding of the grids. This possibility is eliminated by the fact that the 50 percent increase in diameter of wool fibers in formic acid is accompanied by a slight contraction in length (0.5 percent). In addition, evidence given later in this paper indicates that wool fibers in water contain a relatively easily extensible material which contributes 32 percent extensibility to the fibers. Reconciliation of the preceding theory with the results of x-ray examination can, however, be effected if the phase under consideration forms part of the "amorphous" phase of the fiber. If the less heavily cross-linked regions of the fiber are kept in the semi-extended form by thermal agitation, it is easy to understand why they should fail to contribute to the crystalline pattern and hence why the β-keratin remains invisible.

TABLE II

Kind of wool	Stress ($g. \times 10^5$) (per cm^2) f	Immediate extension (%) E	$\frac{E}{32}$	$\tanh^{-1}\left(\frac{E}{32}\right)$
Cotswold	5.05	2.64	0.0825	0.0827
	5.38	9.13	0.2853	0.2935
	6.14	13.2	0.4125	0.4387
	6.95	18.6	0.5813	0.6645
	7.02	19.5	0.6094	0.7080
	8.54	28.4	0.8875	1.410
	10.04	30.1	0.9406	1.743
	10.16	29.6	0.9250	1.623
	11.93	32.7		
	13.11	34.7		
	14.92	35.0		
Australian Leicester	5.79	8.69	0.2715	0.2785
	8.10	26.2	0.8188	1.153
	10.78	32.0		
	14.15	35.8		
	15.96	36.8		
Australian Merino	8.86	29.7	0.9281	1.645
	15.14	36.8		

Attention was next turned to the behavior of the less easily extensible phase in formic acid—the difference curve of Figure 5. A rough estimate of the energy involved indicates that there must be 1–2 percent extension of the phase, even at zero tension. Its maximum extensibility seems to be of the order of 100 percent of the unstressed length of the fiber, but the exact value is uncertain because the fiber broke at 70 percent extension—that is, when the less extensible phase had permitted 38 percent extension. Any error in the value chosen for the limiting extension is, however, without serious effect on the calculated values of ΔG^0 and λ. In the following calculations it has been assumed that the less extensible phase extends from 2 percent to 100 percent according to the type of law illustrated by curve B of Figure 4.

Since

$$\beta = \frac{E - E_1 + 2}{100},$$

it follows from equation (5) that:

$$1 - \frac{E_2}{50} = \tanh\left(\frac{\Delta G^0}{2RT} - \frac{\lambda\lambda'\lambda'' f}{kT}\right), \qquad (13)$$

where

$$E_2 = E + 2 - E_1$$
$$= E + 2 - 32 \tanh(0.42f). \qquad (14)$$

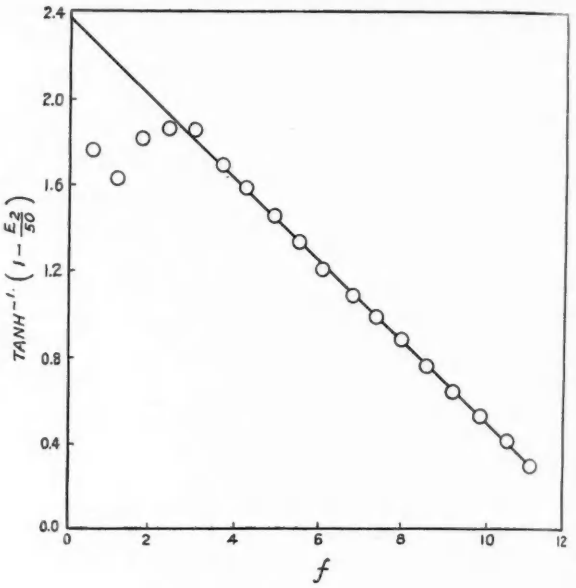

FIG. 6.

Values of $\tanh^{-1}\left(1 - \frac{E_2}{50}\right)$ are given in Table I and, as shown in Figure 6, they give a linear relationship* when plotted against the corresponding values of f, in accordance with equation (13). The intercept is 2.39, giving a value of 2,810 calories/mole for ΔG^0, and the slope of -0.188 indicates that $\lambda = 0.85$ Å. This value of λ is much lower than that found for the first phase, as would be expected if the second phase is more heavily cross-linked than the first. It should, however, be noted that the value of λ for the first phase was calculated by assuming that the area of cross section of each chain is the same as in the crystalline phase.

The value of 100 percent, used in the above calculations as the contribution made by the second phase to the limiting extensibility of the fiber, is only a rough approximation, but any error in the estimate is more likely to be one of deficiency than of excess. Therefore, together with the 32 percent extensibility due to the first phase, the fiber should possess a total extensibility of 132 percent from the unstretched length, or of $\frac{164 \times 100}{68} = 241$ percent from the completely folded form. Since wool fibers are known to be capable of undergoing a reversible supercontraction of about 30 percent,

* Neglecting the first four points, which are seriously affected by small errors in estimating the value of E_2.

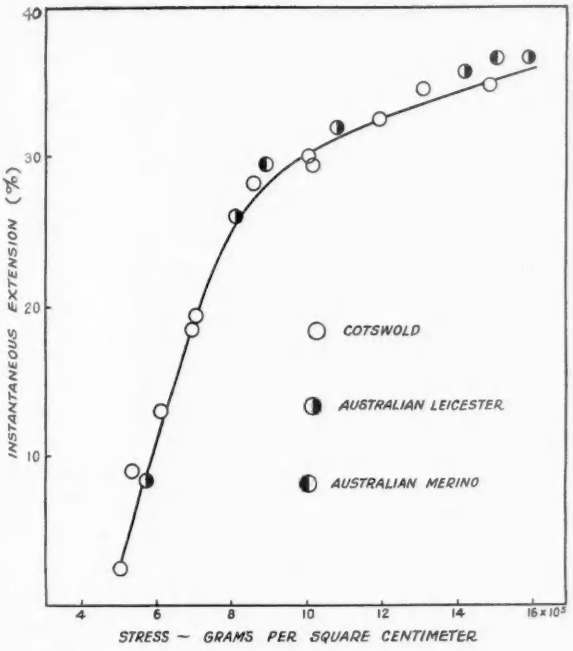

FIG. 7.

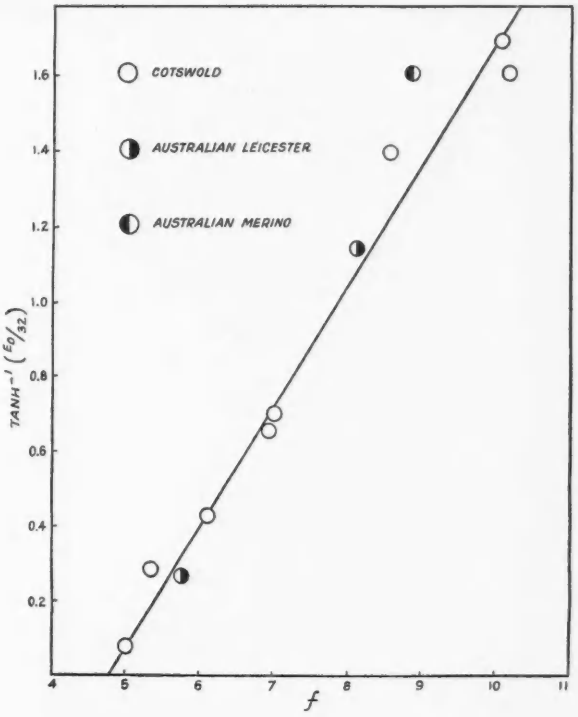

FIG. 8.

while extensions of over 100 percent of the unstretched length have been observed in steam, the above calculated values of extensibility may be regarded as reasonable. Such high extensibility is, however, difficult to reconcile with the small values of λ, unless unfolding occurs in a series of small steps. More detailed examination of the elastic properties of wool fibers in formic acid must be undertaken before the precise nature of the unfolding process can be elucidated and the bearing of these observations on the current conception of the nature of α-keratin can be discussed.

Behavior of Wool Fibers in Saturated Air

The elastic properties of wool fibers in saturated air are very much more complicated than in formic acid because the grids, which are separated in the latter medium, are superimposed and bonded together through hydrogen bonds and van der Waals forces in saturated air. If, however, these extra forces merely shorten the distance through which each unit moves during unfolding and introduce extra energy terms, the general theory should be capable of application to the behavior of the fibers in saturated air. Unfortunately, wool fibers show pronounced creep in saturated air, even under constant stress, and the equations used in this paper become extremely complicated and unwieldy when the time factor is allowed for. The difficulty can be overcome by confining attention to the instantaneous extensions of a series of fibers under different (constant) stresses, obtained by extrapolating the extension-time curves [9]. Data of this type for wool fibers in saturated air at 15° C are reproduced in Table II.

The extension-stress curve is shown in Figure 7 and resembles that of wool in formic acid but displaced to a higher stress before marked extension takes place. In order to discover whether the two curves really have the same form, each extension E was divided by 32 and the value of $\tanh^{-1}(E/32)$ (Table II) was plotted against f. There is a linear relationship between the two, as shown in Figure 8, and the value of λ obtained from the slope of 0.317 is 1.4 Å. As expected, the value of λ is of the same order as, but less than, that derived from the load-extension curve of the same phase of the fiber in formic acid.

The value of the intercept derived from Figure 8 is −1.52, but in interpreting its significance it is necessary to remember that the above extensions are instantaneous and, presumably, adiabatic. An instantaneous extension of 30 percent can give a rise in temperature of over 8° C, but if the subse-

quent gradual cooling is corrected when the extension-time curve is extrapolated over a period of about 1 minute to zero time there is no change in entropy and $\Delta G^0 = \Delta H^0$. The value of ΔH^0 derived from the intercept is 1,740 calories/mole.

When wool fibers are transferred from formic acid to water, their change in length is negligible. The equilibrium constant for the process of unfolding must, therefore, be the same in the two media, and, since ΔG^0 is zero in formic acid, it must also be zero in water for isothermal extensions. Hence

$$\Delta H^0 - T\Delta S^0 = 0$$

and $\Delta S^0 = 6.0$ entropy units/mole. This value is interesting because it is of the same order as the entropies of melting of crystals which owe their cohesion to hydrogen bonds.

During the extension of wool fibers in water, hydrogen bonds between peptide groups may be replaced by hydrogen bonds between water and peptide groups. The term ΔH^0 is therefore composite and the above value of 1,740 calories/mole represents the difference in bond energy between two types of hydrogen bonding. At low relative humidities, therefore, the energy required to unfold the peptide chains must be greater than in water, and the load-extension curve will be displaced to higher loads. It should be possible to obtain a value for the intrinsic strength of the hydrogen bonds between peptide groups from the load-extension curve of a dry fiber, and the difference between this value and the one derived from the load-extension curve of the fiber in water will provide an estimate of the water-keratin bond energy.

Analysis of the load-extension curve of the less easily extensible phase of the fiber in saturated air is more difficult because the activation energy for the unfolding process is large and equilibrium is never attained. The failure of wool fibers to reach an equilibrium extension, even under constant stress, is in good agreement with the following conception of the way in which decay of tension takes place in fibers at constant extension. When the fiber is first stretched, the easily extended, loosely cross-linked phase is overstretched and the highly cross-linked phase is understretched. During relaxation, the first phase contracts and the second phase extends so as to reduce the total free energy to a minimum. The rate of approach to equilibrium increases with rise of temperature, but a truly reversible equilibrium is never attained because disulfide-bond breakdown and linkage-rebuilding processes intervene.

Despite its simplicity, the preceding theory affords a satisfactory explanation of the elastic properties of wool fibers in formic acid and saturated air, including relaxation processes and reversible supercontraction. An attempt is now being made to develop the theory still further by using it to interpret the behavior of ordinary and fully reduced fibers at different humidities.

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Certain Variations in the Structure of Wood Fibers

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THE FINE STRUCTURE of plant-cell walls varies not only from one type of plant to another but also within a given species or variety and even within a given plant. Natural cellulose fibers of commerce, such as cotton, flax, abaca, and wood pulp, are composed of plant cells. The physical properties of fibers are dependent upon their cell-wall structure and the way in which they are woven together to form fabrics. Much can be learned by comparing the properties of the different fibers which tend to compete with each other. An attempt was made in this study to ascertain the structure in the different regions of the fiber. Wood cells were used so that each cell could be held in position throughout the study. Additional studies are needed on cotton and other single-cell fibers, using microtechniques.

Preston [1] made certain observations on the nature of the spiral structure of the cellulose in plant tissue, stating that there was but one spiral which varied from one part of the cell to another. Bailey and Berkley [2] observed that there were two or more separate spirals in the various layers of the cell wall and that the pitch and direction of each was independent of the other. The observations and illustrations presented here confirm these conclusions and show, in addition, variations within a given spiral. The conclusions reported here are in harmony with the data presented by Caldwell and Lock-Horovitz [3] and tend to explain the wide variations in fiber structure shown by the x-ray patterns in their report.

Materials and Methods

The materials used in this study consisted of specimens of woody stems and roots. The microscopic techniques used were those associated with the ordinary light microscope and the petrographic microscope. The specimens were prepared either by simple extraction to remove terpenes or by delignification of small blocks of wood. The wood rays of certain samples were carefully removed, care being taken to disturb as little as possible the arrangement of the fibers.

The Kα radiation from a copper cathode x-ray diffraction tube was used to obtain the diffraction patterns.

The patterns were made by passing the x-ray beam: (1) perpendicular to the long axes of the fibers and parallel with that of the wood rays (radial); (2) perpendicular to both the long axes of the fibers and that of the wood rays (tangential); (3) perpendicular to the long axis of the fiber and at a 45° angle to the long axes of the wood rays—that is, diagonal to both the radial and tangential positions; and (4) parallel with the long axes of the fibers. (See Figure 1.) If the x-ray beam were passed in the radial direction it would pass through the tangential walls perpendicular to the plane of the concentric laminae of the cell and parallel with

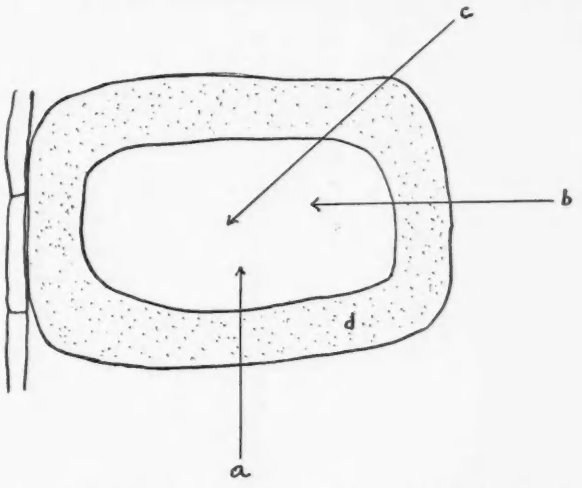


FIG. 1. *Diagram of cross section of wood fiber indicating the positions: (a)—radial—that is, perpendicular to the fiber axis and parallel with wood rays; (b)—tangential; (c)—diagonal to (a) and (b); and (d)—parallel with the long axis of the fiber. The small cells on the extreme left represent wood ray cells.*

*National Cotton Council of America.
†Signal Corps, U. S. Army.
Both authors were members of the Bureau of Plant Industry, Soils and Agricultural Engineering, U. S. Department of Agriculture, when this work was done.

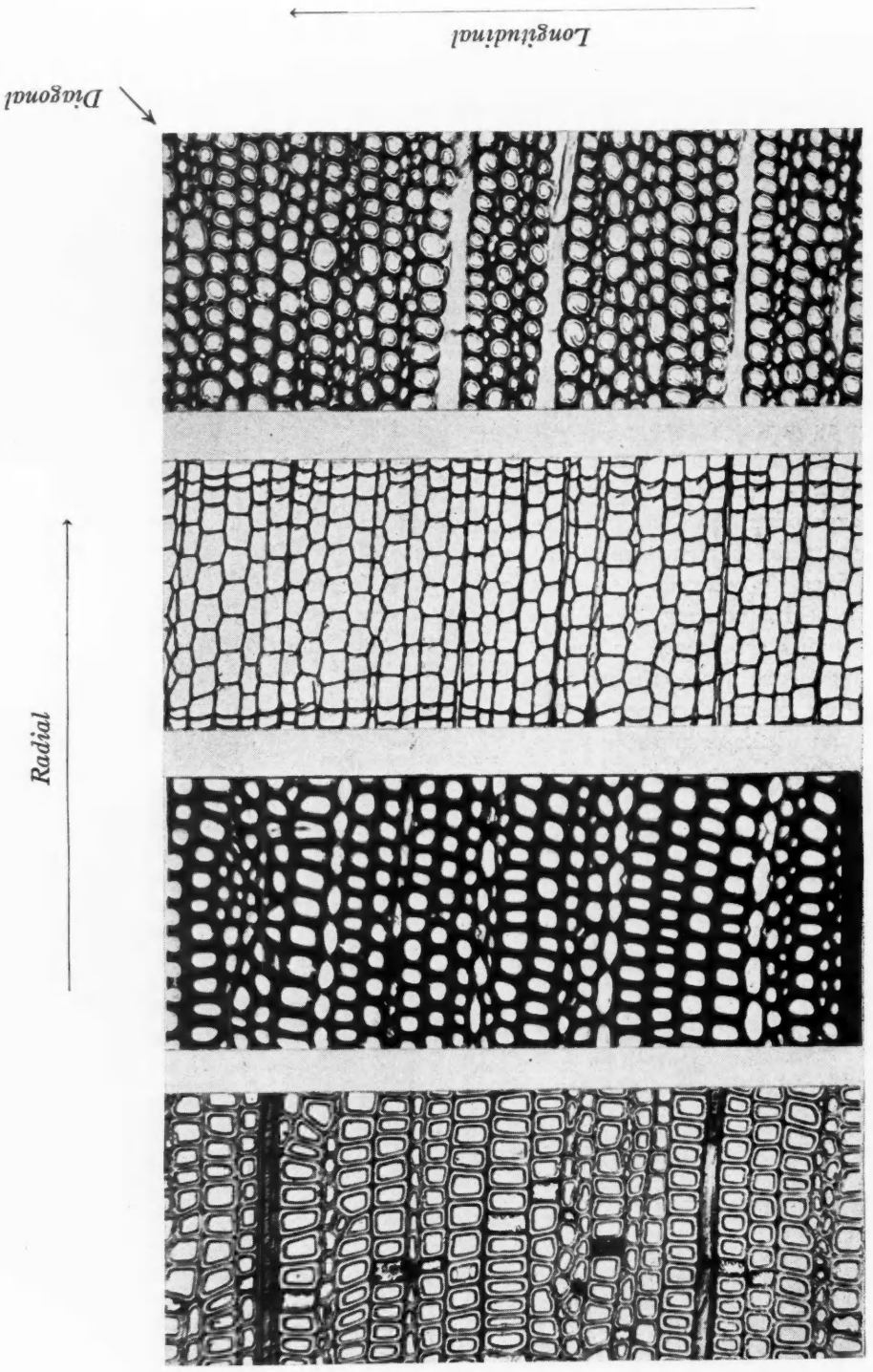
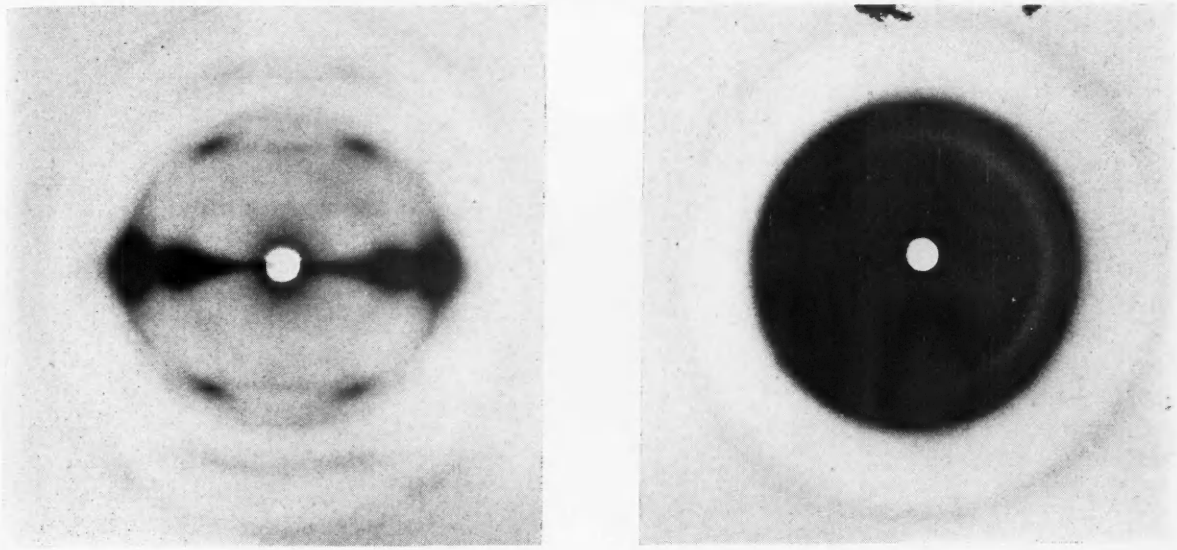


FIG. 2. *Cross sections of fibers and other cells from stems and roots used to obtain the x-ray diffraction patterns reported: A—Sequoia, late wood, 145 ×; B—Trochodendron, late wood, 145 ×; C—Taxodium root, 55 ×; and D—Pinus "rotholz" (compression wood) 145 ×.*



A B

FIG. 3. *X-ray diffraction patterns from late wood of* Pinus palustris: *A—radial; B—parallel. In all x-ray patterns the specimen position was vertical.*

the laminae in the radial walls. The opposite would be true for the patterns obtained by passing the x-ray beam in the tangential direction—that is, the x-ray beam would pass perpendicular to the plane of the cellulose laminae in the radial walls and parallel with them in the tangential walls.

Results

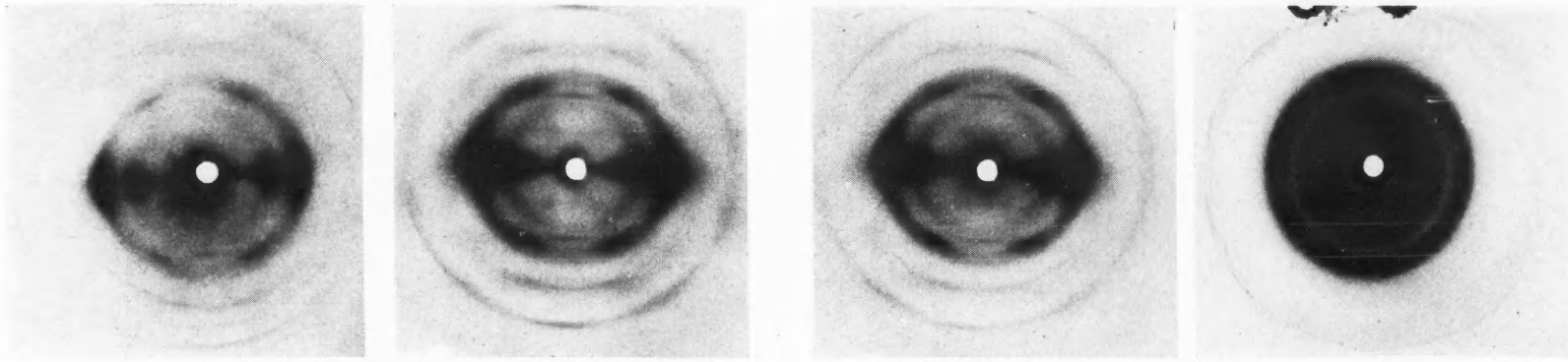
Cells of woody plants have many properties in common and frequently appear to be very similar when observed under an ordinary microscope. If they are stained with dyes, however, differences in structure as well as shape can frequently be brought out. Extraction and chemical purification bring out still other differences.

One of the outstanding differences between plant fibers is the fine structure recorded in x-ray diffraction patterns. These highly complex recordings may be variously interpreted to give information on the general over-all structure, particularly the crystalline phase of the cellulose in the cell walls. Accepted crystallography makes possible rather precise space-lattice measurements, but the orientation and imperfections of the crystallites may lead to false interpretations of the size and general nature of basic units if other data are not considered.

The size, shape, and cell-wall thickness of the samples used varied widely (Figure 2). In general the cells were rectangular; however, the "rotholz" (compression wood) cells in one sample of Pinus were more or less circular. These samples were no doubt all different in chemical constituents.

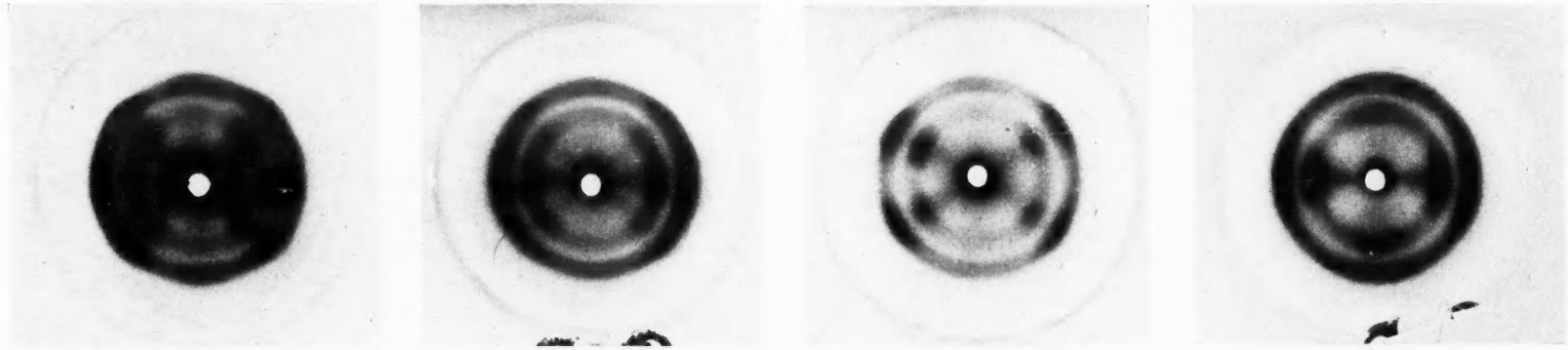
X-ray diffraction patterns obtained from the fiber of the summerwood of *Pinus palustris* (Figure 3) show by the short arcs (most prominent spots resulting from 002 lines) that the cellulose lies essentially parallel with the fiber axis. Similarly, the patterns from Trochodendron (Figure 4) show little difference in spiral arrangement of the cellulose in the tangential and radial walls. It is not possible to establish the exact angle of spiral in these samples by the x-ray method, since the maxima from opposite walls blend, but the microscopic observations indicated an angle of spiral not in excess of 10° to 14° between the long axes of the crystallites and that of the fiber.

The pattern obtained by passing the x-ray beam "parallel" with the long axis of the fiber (extreme right in each figure) is a typical "Debye-Scherrer ring pattern" with essentially equal distribution of photographic intensity around the rings diffracted from the various planes in the crystallites. This pattern may be interpreted to indicate that either the corners of the cells diffracted equally with the tangential and the radial walls or that the cellulose crystallites are more or less randomly arranged



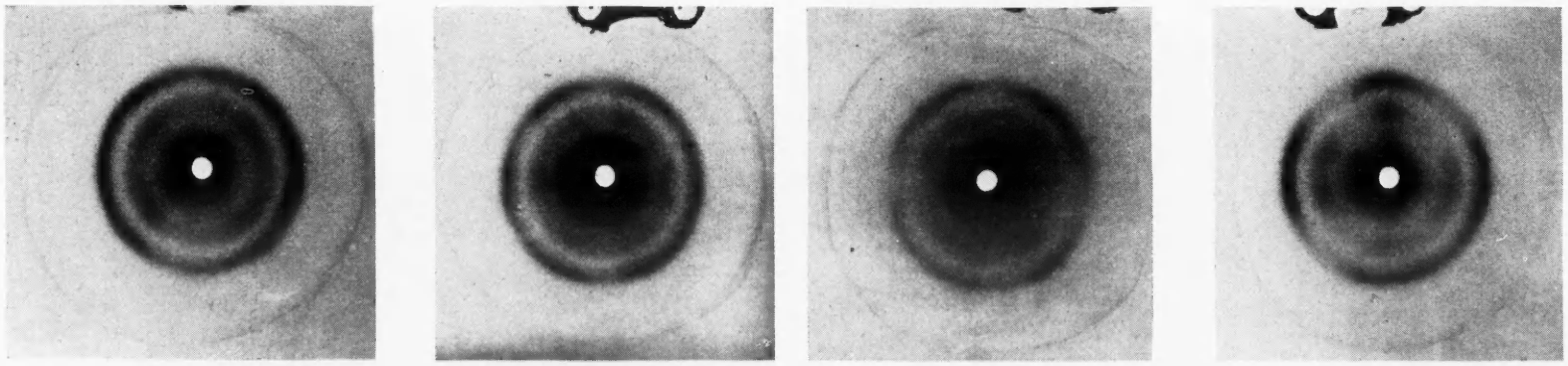
A B C D

FIG. 4. *X-ray diffraction patterns obtained from a sample of late wood from Trochodendron: A—x-ray beam passed perpendicular to long axis of wood fiber and parallel with long axis of wood rays, radial; B—perpendicular to both the fiber and the wood rays, tangential; C—perpendicular to the fibers and 45° to the long axis of the wood rays, diagonal; and D—parallel with the fiber axes.*



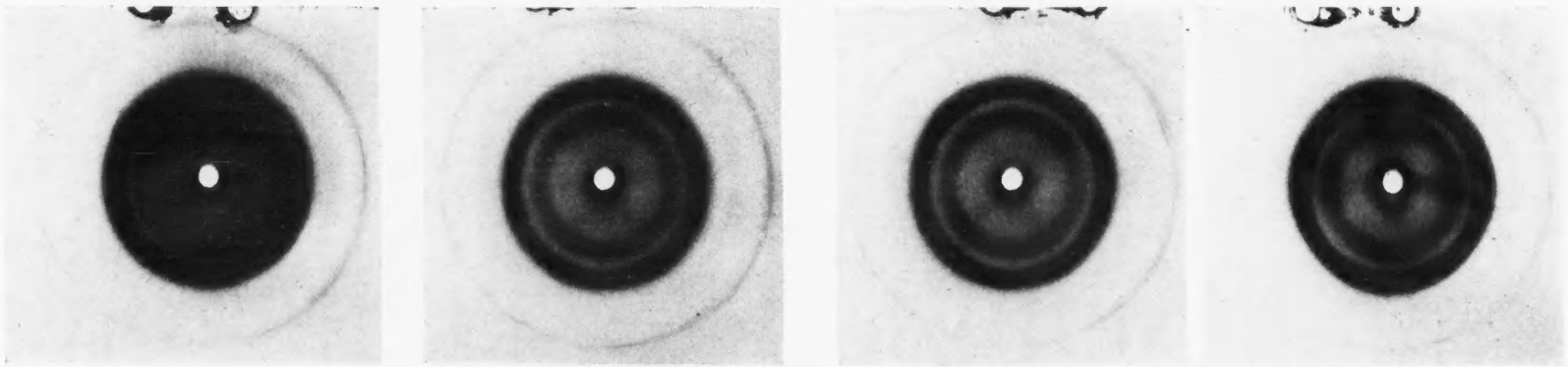
A B C D

FIG. 5. *X-ray diffraction patterns from Sequoia: A—radial; B—tangential; C—diagonal; and D—parallel.*



A B C D

FIG. 6. *X-ray diffraction patterns of Taxodium root: A—radial; B—tangential; C—diagonal; and D—parallel.*



A B C D

FIG. 7. *X-ray diffraction patterns from "rotholz" (compression wood) of Pinus: A—radial; B—tangential; C—diagonal; and D—parallel.*

(rotated in all positions) with respect to their lateral axes.

It was extremely difficult to set the fibers of Pinus (Figure 3B) nearly enough parallel with the x-ray beam to obtain a symmetrical ring pattern. One side was invariably more dense than the opposite side but there was no indication of selective orientation.

The cross sections of the cell of Trochodendron (Figure 2B) used in obtaining these patterns are similar in contour to those of Sequoia (Figure 2A), which gave entirely different results.

The x-ray pattern taken in the "radial" direction from Sequoia (Figure 5A) shows a spiral arrangement of the cellulose. Two pairs of maxima appearing at approximately 50° and 230° and at 130° and 310° indicate a spiral in the tangential walls of the fiber at an angle of 40° to the axis of the fiber. Microscopic examination showed that these maxima were obtained from the cellulose in the central layers of the secondary thickening.

The slight maxima at 0° and 180° indicate transversely arranged cellulose apparently in the primary wall and in the outer and inner layers of the secondary thickening. The maxima at 90° and 270° indicate that there is also crystalline cellulose essentially parallel with the long axis of the fiber. This is more strongly indicated in the pattern from the tangential direction (Figure 5B) in which the maxima at 90° and 270° are pronounced. No trace of maxima at these points can be found, however, in the pattern obtained from the diagonal position. No immediate explanation of this is available, but it may be due to the wood rays or to the pits in the fiber walls. The pattern from the radial position shows the spiral of the cellulose in the tangential walls, that from the tangential position shows the orientation of cellulose in the radial walls, and that from the diagonal position shows certain elements from each of these walls. It was observed that the cellulose spiral in the tangential walls was about 40°, and that of the radial walls was 35°. The pattern obtained from the diagonal position indicated that the pitch of the spiral was approximately 30° to the fiber axis. There were two pairs of maxima at 90° to each other in the pattern from the parallel position. One pair of maxima resulted from the tangential walls and the other one from the radial walls.

Figure 6 consists of the x-ray diffraction patterns obtained from the fibers of Taxodium root. The maxima are not so distinct as in the patterns from Sequoia (Figure 5). The maxima which do occur indicate that the spiral in the tangential walls (x-ray pattern from radial position) is 42.5° and that there is cellulose arranged parallel with the fiber axis. The patterns from the radial walls (tangential pattern) show a spiral of 60° and cellulose essentially parallel with the long axis of the fiber. The diagonal pattern shows a spiral at approximately 50°, which would be a mean of the other two spirals. The cellulose parallel with the fiber axis does not show clearly in this pattern. The pronounced maxima in the pattern obtained when the x-ray beam was passed parallel with the long axis of the fiber were from the tangential and radial portions of the cell walls, and here, as in Sequoia, the corners of the cells diffracted little if any. Note the broad maxima at the right and left. There is a tendency for three sets of maxima to occur, one from the tangential walls and two from the radial walls. The zigzag position of the radial walls, as shown in Figure 2C, gives two sets of planes, each of which, presumably, is responsible for a faint pair of maxima which blend more or less to give the broad arcs.

The x-ray diffraction patterns obtained from a specimen of "rotholz" from *Pinus ponderosa* L. are illustrated in Figure 7. All of these may be considered Debye-Scherrer ring patterns, with only slight maxima indicated. The slight maxima in the patterns from the radial and tangential positions show the positions of the spiral in the tangential and radial walls, respectively. The spiral in the tangential walls was at an angle of about 47.5° to the fiber axis and that of the radial walls was at an angle of approximately 56° to the fiber axis. The pattern for the diagonal position shows an angle of about 55°. The pattern from the parallel position also shows a Debye-Scherrer ring but slight maxima are present. Since the fibers in this sample were "rotholz" cells, the secondary thickening was almost cylindrical (see Figure 2D). The slight maxima that occurred may be due to wood rays or to cells that were not round and therefore not of the "rotholz" type.

Discussion

An x-ray diffraction pattern, as used in these studies, does not necessarily show, clearly enough to be measured, all of the crystalline cellulose present in a sample. It is also difficult to understand and interpret all of the variations that can be measured in the patterns. It is quite clear, however, that the

patterns obtained from the various samples represented here are different, and a careful microscopic study of the tissue was made to supplement the x-ray method in arriving at the interpretations given.

The patterns obtained from Trochodendron are interpreted to show that the major portion of the crystalline cellulose lies more or less parallel with or at a small angle to the long axis of the fiber. The small portion of the cellulose contained in the primary wall or in the outer or inner layers of the secondary thickening was found by the use of a microscope to be arranged essentially perpendicular to the fiber axis. The patterns from Sequoia, on the other hand, are interpreted as showing that there are at least three spirals. Preston [1] failed to observe evidence of different spirals in the materials he studied. The patterns from Taxodium root also indicate the presence of more than one spiral and, as in Sequoia, show that the spirals on the tangential walls have a different pitch from those on the radial walls. The patterns from the Pinus sample show a more or less random distribution, due either to a multiplicity of layers each having a different spiral, or, as appears more likely from the microscopic study, to a tendency for radial arrangement of the cellulose in the central layer of the secondary thickening of the cells making up this sample and the very low-pitched spirals in the outer and inner layers of the secondary wall and in the primary wall of the individual fibers. The presence of two or more spirals can be detected by the faint maxima.

The patterns obtained by passing the x-ray beam parallel with the long axes of the fibers are difficult to interpret. The pattern from Trochodendron is a uniform ring pattern showing no preferred orientation, whereas those from Sequoia and Taxodium root show pronounced maxima. The position of the maxima indicates that either the corners of the cells in fibers with a pronounced spiral structure contain little or no crystalline cellulose, or, if present, it is dominated by the plate-like portions of the cell forming the more or less flat sides. If crystallites exist in the corners of the cells, they are rotated so that they do not register in the pattern. The lack of evidence of crystalline cellulose in the portion of the pattern from the corners of cells with a pronounced spiral structure is further indicated by the fact that these fibers swell and shrink in diameter with changes in moisture content. Since crystalline cellulose does not change in dimension with changes in moisture content, it would be difficult to explain how such cells, if the spiral was continuous around the corners, could swell and shrink. Furthermore, the spiral is frequently at a different angle on the adjacent walls.

Acknowledgment

The specimens used in this study were prepared by Dr. I. W. Bailey of Harvard University, to whom the authors wish to express their appreciation.

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Moisture Determination in Textiles by Electrical Meters: Part I

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Contribution of the Textile Research Institute and of the Department of Chemical Engineering, Princeton University, Princeton, New Jersey

Abstract

Results are reported from a study of two electrical moisture meters adaptable to bulk textile materials. Calibration curves at a single temperature are reported on white, scoured, wool yarns.

Accuracy of the meters is dependent upon a calibration against an independent measurement of moisture. A modification of the loss-in-weight oven method was used as reference.

Precision for both ovens and meters is based upon statistical analysis of groups of data. For instruments, in the best cases, the value was approximately 0.25 percent with the ovens at 0.40 percent moisture, both figures based upon two-sigma limits, sampling variance having been removed.

Introduction

Moisture analysis is important in the textile industry as a basis for purchase and sale as well as for process control. Analyses, usually made by an oven, are often unsatisfactory, particularly in control work, because of the associated time delay. An instrument which would rapidly and accurately determine the moisture content of textile materials would offer convenience to the operator and economy to management. A study of representative types of moisture meters, which have been advertised as suitable for the textile industry, was undertaken by these laboratories to evaluate the variables which are met in factory operation, such as type of material, its previous history, dye treatment, and temperature. While the study includes instruments adaptable to both bulk and fabric, the current report will deal with only two meters suitable for bulk materials and will be limited to a single textile material at a single temperature. Statistical methods used to evaluate precision will be summarized. Subsequent papers will present the remaining tests on these and other meters.

The results and comparisons which are presented are not intended as a recommendation or disapproval of any specific instrument, but they are suggested as a logical means by which an evaluation may be made and they are expected to indicate general operating characteristics of moisture meters.

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Types of Meters

Although a few moisture meters (notably the Shirley and the Fielden) were designed specifically for textiles, the majority were developed primarily for other industries. Their basic principle, however, is common: the water content of a substance is determined by means of either direct-current resistance, alternating-current impedance, or alternating-current capacitance, since a small change in moisture greatly affects these properties. The form of the meter is often dictated by the material for which it was designed. For example, an A.C. meter used widely in the grain industry consists of a cell into which the substance under test is packed; an adaptation of this meter in the textile industry may be used on bulk material such as sliver, tops, yarn, etc. A second meter, which had its origin in the paper trade, is of the D.C. form, consisting of parallel plates which are brought into contact with a sheet. This design is suitable for fabric or other sheet forms but may be adapted to bulk material. A variation of the parallel-plate type using alternating current of radio frequency has recently been announced in which passage of the textile through the electrical field is sufficient to obtain a reading; it is unnecessary to make physical contact with the plates. Obviously, this de-

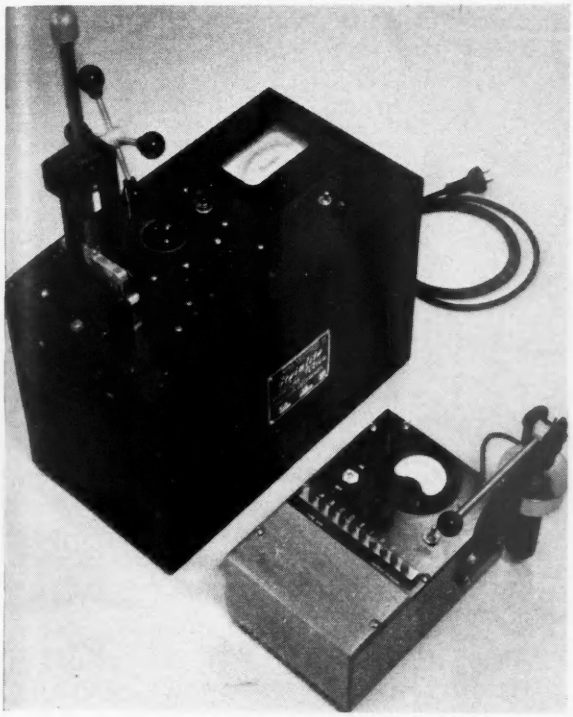

FIG. 1. *Steinlite and Hart moisture meters.*

sign is of particular advantage when the textile material is in motion.

Other meters have two or more prongs or spaced electrodes which penetrate the textile material, and these are used for testing heavy felts or the contents of bales; still others employ rotating cylindrical contactors for moving materials such as warps from the slasher driers.

Meters Tested

The present report discusses two meters, one each from the D.C. and A.C. fields, but their selection is without implication of perfection of either of these in its field. A third meter, also designed for bulk material, was received too late to be included in this investigation, but tests on it are now being conducted and will be reported subsequently. Although also in the A.C. field, its circuit indicates the utilization of a different moisture-sensitive electrical property from that involved in the A.C. meter being discussed here.

Operation

Since complete operating instructions accompany each instrument, only the basic essentials of test methods need be described. The Hart Moisture Meter, illustrated in Figure 1, is the D.C. instrument used. It is the standard instrument supplied for wool analysis and, except for a static suppressor, is without special design features. The sample to be tested is placed between two flat, circular electrodes. Depressing the lever handle compresses the sample against a heavy spring, insuring electrical contact between sample and electrodes. A meter reading of zero is obtained through use of a variable resistor. The handle is released and the sample removed, after which the range buttons are depressed in sequence (introducing standard resistances into the circuit) until a resistance equivalent to that of the sample is found. Usually, one resistance will give a reading which is slightly low while the adjacent one is slightly high. Interpolation between the moisture contents which these buttons represent gives the value sought. This instrument must be calibrated against some standard, such as oven moisture.

The Steinlite Moisture Tester, also illustrated in Figure 1, is an A.C. meter. The calibrating condenser is adjusted prior to charging the cell to bring the indicating needle to an arbitrary point near the middle of the linear scale. For this instrument it is necessary to weigh out the sample of bulk material and insert it into the cell, after which a Lucite plunger is driven downward by a rack-and-pinion mechanism, compressing the material into a definite volume limited by a mechanical stop. When the cell is packed, the appropriate range button is depressed and a meter reading, which is essentially a measure of impedance, is obtained. This reading is converted to moisture content through a calibration curve.

Sample Preparation

Since the Steinlite is capable of measuring bulk material only, all tests which form the basis of this report were made on a white, scoured, wool yarn, size ⅛ 50's. The standard desorption samples were prepared by soaking the yarn in distilled water, in which a few drops of Tergitol had been added to facilitate wetting, and rinsing several times in distilled water, the last distilled rinse water being discarded after use on a single sample.

Because it was thought that sample history might have an effect upon the meter analysis, special sorption and desorption samples were prepared in a slightly different fashion. In this case, all of the material was heated at 100°C for 2½ hours to re-

move the bulk of the moisture. One-half was stored in a desiccator until ready for use. The other half was either sprayed with approximately 50 percent of its own weight of water and placed in sealed cans for several days to permit all the fibers to become uniformly wet, or allowed to remain in contact with moisture-saturated air.

When ready for test, both sorption and desorption samples were exposed under conditions of controlled temperature and humidity.

Oven Analysis

As generally realized, the oven method of analysis is not a sound standard, since the type of oven used, the ambient conditions, and other factors such as volatilization of materials other than water affect the calculated value. But, although true moisture content is not necessarily determinable by the oven method, this method was convenient for the current tests, particularly since a large number of samples with no known volatiles (other than water) were involved and speed was not essential. In order that the reference standard used for these tests be free from some of the objections often present, all analyses were made in the same oven, a natural convection type, in a conditioned room at $70° \pm 1°F$ and 70 ± 1 percent relative humidity. After the samples had been heated for 1½ hours at 110°C, the oven door was opened and the moisture-laden air was swept out by means of a fan; drying was continued an additional hour; samples were cooled in a desiccator and weighed.

Conditioning Methods

It seemed desirable to make the basic calibration curve from samples which were conditioned to equilibrium in order that variation between the meter samples and those analyzed in the oven be held to a minimum. Initially, measurements were made only after test strips, inserted in the skeins being conditioned, showed essentially no day-to-day change in weight. True constant weight was never obtained; even after a period of several weeks, both sorption and desorption samples showed very slight, but significant, changes. However, the major portion of the gain or loss in weight occurred within a matter of hours, and it became evident that after two or three days the change was so gradual that samples could be used for both oven and meter tests with reasonable assurance of equality of moisture content. The term "equilibrium samples" in this report refers to samples conditioned for at least this minimum period.

A long time is required to get data by equilibrium testing even under ideal conditions. If dry and wet samples are exposed to a conditioned atmosphere and a series of readings is taken as the samples either gain or lose moisture, an entire calibration curve might conceivably be prepared in a relatively short time, although, as will be subsequently noted, these data cannot be used for evaluating the precision of the meter reading. Early work in these laboratories and a comparison of this work with similar studies elsewhere indicated that such a "grab sample" technique gave results which differed from those obtained with samples at approximate equilibrium, but the difficulty might be due to the failure of moisture in the oven sample to correspond to that in the sample taken for meter test. When the technique was refined, the results from the two methods were comparable, with the exception of the first few sorption points, where moisture change is most rapid.

The revised technique requires allotting a predetermined time interval for the Steinlite tests, with the oven sample being taken at the midpoint of this time period. Since the Steinlite sample is out of contact with the conditioned air during the test period, the oven sample skein is also removed from the conditioned air for the same period of time by placing it in an empty desiccator, and it is otherwise given essentially identical treatment with respect to exposure time. Since the test sample for the Hart calibration is not removed from the conditioned atmosphere for more than a few seconds, it is unnecessary to give the corresponding oven sample any storage treatment.

Preliminary Tests

The Hart meter is sensitive to sample thickness, although this effect must not be considered a result of spring pressure variation. It is therefore important to maintain uniform thickness among samples tested, and since the yarn used was of constant size, test samples were prepared by taking 10 strands, 8 inches long, holding them so that a 5-inch length was exposed, and imparting one complete twist.

For the Steinlite, tests were made first with yarn in skein form, second, with strips of the approximate width of the cell, and, last, with strips less than an inch in length. Regardless of the length of sample used, the results were consistent. The uncut yarn

could be packed in at least one-half the time required for the other methods and was more convenient in handling; consequently, in all later tests continuous lengths of yarn were used.

The Steinlite readings are quite sensitive to density; that is, the reading constantly changes as the sample is compressed if the weight of yarn is kept constant, and, similarly, the reading will vary if the weight of yarn used is changed but the volume of the testing cell is kept constant (*i.e.*, using the same mechanical stop). However, if the length of the mechanical stop is varied as the weight of the charge is changed so that the same density is maintained, the meter reading is independent of the amount of sample charged. The variations due to density changes are least in the region of high density, and so a weight of 600 grains (38.9 grams) with a stop of 3 inches was adopted as a general standard. Under these conditions, the volume of the test cell is 83.9 cubic centimeters, and the electrodes are 61 percent covered by the material under test.

There appears to be a small packing effect when the pressure is applied, released, and reapplied, the readings ultimately becoming constant. However, within a reasonable time limit of several minutes the initial reading does not vary while the sample is under pressure. Hence, any packing effect appears to be due to the repeated application of pressure rather than to a continuous application. The results reported in this paper are for the first application of pressure.

It was thought that better results might be obtained if the Steinlite were always operated at the same pressure on the sample rather than under conditions of constant volume (density). A calibrated spring device was constructed to insure duplication of pressure, but a comparison of the constant-pressure method with the constant-density method failed to reveal any superiority of the former and it required a more complicated design; it was therefore discarded and the constant-density method was retained for all subsequent tests.

Analysis of Data

All data were analyzed statistically. Whenever a curvilinear correlation is reported, it may be inferred that statistical evidence for such curvilinearity exists. The choice of coordinates was also dependent upon statistical considerations; if the variance of the quality measured was independent of the magnitude of the variable, normal coordinates were used; but if the variance increased with numerical magnitude, a logarithmic scale was chosen. Under these circumstances, the "two-sigma limits" may be adequately presented as parallel to the calibration line.

The primary standard, oven analysis, is actually subject to the variance from the sample plus that inherent in the oven method. Sample variance will also be present in the instrument and it may reasonably be expected that this method of analysis, too, will be subject to some uncertainty. To arrive at a value for the instrument alone, one must remove that portion of the total variance in the meter reading which is assignable to sampling variance. The two-sigma limits which are reported have been calculated with this principle in mind; thus 95 percent of all instrument readings should fall between these limits for a given value of moisture when sample variation has been excluded. The two-sigma limits which are shown as dotted lines are the limits described.

The Basic Calibration Curve

For the basic studies, a temperature of 70°F was chosen for the convenience of the operator. Equilibrium studies served to determine base points, with grab-sample runs on sorption samples being used to obtain points between the equilibrium values. As will be seen from Figure 2, all of the equilibrium points lie quite close to the calibration lines when the logarithm of the Steinlite reading is plotted against the moisture content by oven analysis. Some, however, fall outside the two-sigma limits since the latter show precision of the instrument alone with sample variance removed.

The basic calibration curve for the Hart meter is shown in Figure 3. It will be noted that, unlike the Steinlite curve which is plotted semi-logarithmically, this plot utilizes normal coordinates for both axes, all Hart data being converted to equivalent *E*-scale readings, to permit graphical representation. Two major lines are shown in Figure 3; one refers to standard samples and the other to special samples, previously described. Thus the effect of sample history is clearly demonstrated. It can be shown by statistical methods that the Steinlite readings are likewise affected by sample treatment, but the differences are not so pronounced.

It would seem that the resistance, and thus the Hart reading, would increase very rapidly in the low-moisture region and decrease sharply in the high-

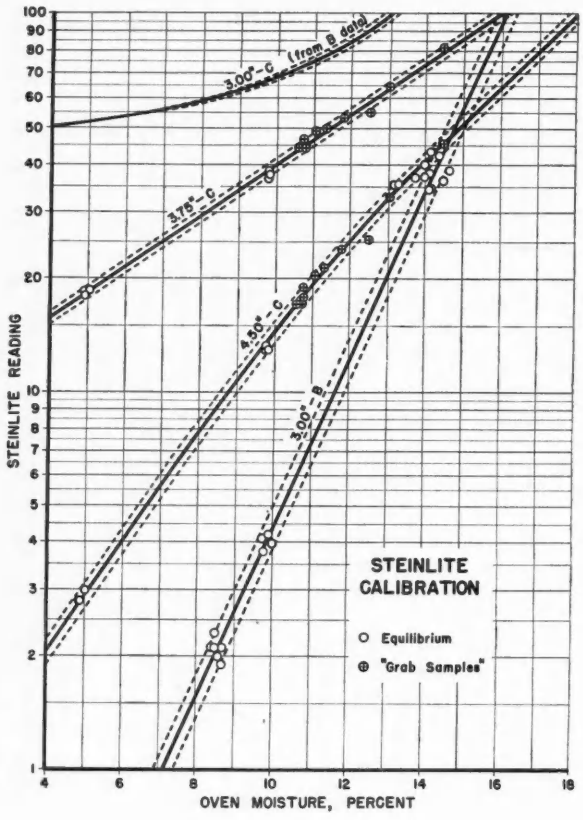

FIG. 2.

moisture range. If these expectations were realized, the calibration curve should logically be of hyperbolic form. The straight line may be explained by the fact that successive resistances are not simple multiples but follow an exponential type of relationship.

Although the calibration curves so far presented for the two instruments are linear, it should be re-emphasized that this result is fortuitous in the case of the Steinlite, for under different experimental conditions its calibration is distinctly curvilinear, as will be subsequently noted.

Relationship Between Steinlite Scales

Reference has been made to the necessity of converting all Hart readings to a single scale for purposes of plotting a continuous calibration curve. It is possible to make these conversions if the unbalance of the circuit as determined by the galvanometer reading is continuously variable from button to button. The design of the Hart meter permits these interconversions, and readings plotted were arbitrarily chosen as those which would result if the *E*

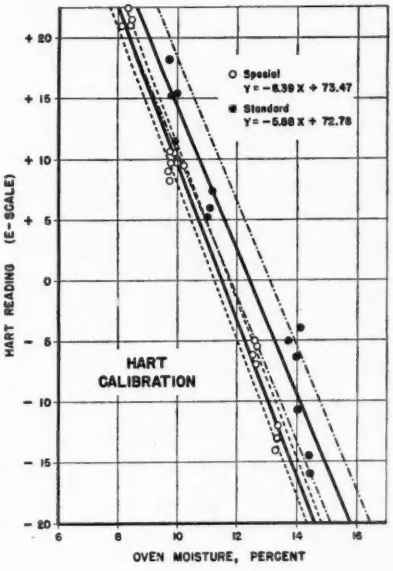

FIG. 3.

button were depressed regardless of which button gave a near-zero reading. Under this system, negative as well as positive readings can be obtained. Since variation in voltage caused either by battery ageing or change in the vacuum-tube activity will affect the relationship between Hart scales, this method of calibration is not recommended by the manufacturer for regular use, the method of matching resistances to balance a bridge circuit being preferred. In the current study this limitation was recognized; hence battery voltages were checked before each test and readings were taken with several buttons and these readings were cross-checked for equivalence.

No such relationship exists between the buttons on the Steinlite. While the ranges overlap, they do not represent a continuous function. In general, readings from 100 to about 50 on one scale have counterparts on the next lower scale from 0 to about 20, as is indicated by the curves shown in Figure 4. Some care must be taken, therefore, to adjust the experimental conditions so that all samples can be analyzed by using a single-range button; otherwise, more than one complete calibration curve must be determined.

The calibration curve can be changed either in form or instrument range by varying the density (mechanical stop size or weight of charge), as is evident from the results plotted in Figure 2, in which the *C*-button calibration curves are either concave downward or upward or, as is the case with the 3.75-inch stop, straight.

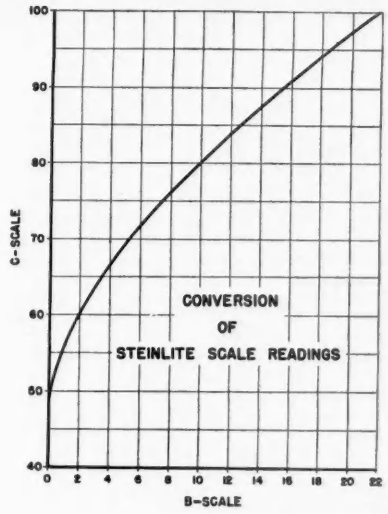

FIG. 4.

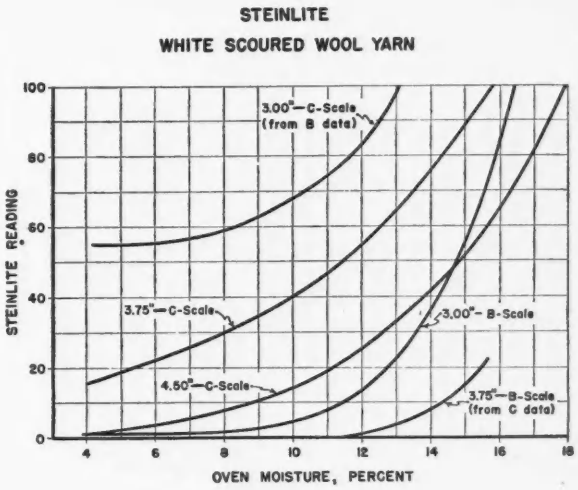

FIG. 5.

Figure 5 is a replot of the least-squares correlation lines presented on normal coordinates. The two-sigma limits would not be parallel to the calibration line because of the change from the semi-logarithmic coordinates but diverge as the moisture increases.

Conclusions

Basic calibration curves for the Steinlite and Hart moisture meters have been presented in this paper, but it is important to note that the user of either of these instruments should calibrate for the material to be tested under the normal temperature conditions of the test. Such a calibration curve can be rather quickly and accurately prepared by the grab-sample technique if the lower portion of the curve is substantiated by adding equilibrium samples. The Hart meter must be used with samples of constant thickness to insure reproducibility of results. The Steinlite meter must be used with proper regard for weight of material and density (that is, the stop employed) inasmuch as these factors determine the type of calibration curve and its range of usefulness. The experimental work on these instruments indicates that, in conjunction with a carefully prepared calibration curve, they will measure the moisture content of similar material with an accuracy comparable to that of an oven, the range of values for the Steinlite as used in this work being ± 0.23 percent moisture. The indicated variance for the Hart differs with the two sample types; the difference probably is due to sampling error and thickness changes. In the better case, the range was ± 0.28 percent moisture, similar to that of the Steinlite, while the poorer results showed limits of ± 0.76 percent moisture.

Acknowledgment

The authors wish to express their appreciation to the Hart Moisture Meter Company and the Seedburo Equipment Company, distributor of the Steinlite Moisture Tester, for the loan of the instruments tested, and to the James Lees and Sons Company for providing the wool; they are particularly indebted to Prof. John W. Tukey of Princeton University for his continued interest in their problems and for his suggestions of methods to be employed in the statistical interpretation of the experimental results; they are also grateful to Miss Angela Bornn for her work in the early stages of the investigation.

APPENDIX

Statistical Methods

General Character of Calibration Lines

All calibration lines are plotted in such a manner that equal variances will be obtained at different points on the scale. In all cases, X refers to percent moisture content. With the Hart meter, Y will refer to readings on the meter converted to the equivalent reading which would be obtained with a single resistance, that marked E. For the Steinlite, variances are uniform over the meter range only when

TABLE I. SUMMARY OF V_O AND V_I CALCULATED FROM EQUATIONS (4) AND (5)

n	Average moisture (%)	β	V_O	V_I	V_I/β^2
Hart Samples, E-Scale Readings, 70°F, Desorption, Standard Preparation					
4	9.96	−5.88	0.00000	7.25	0.2038
3	11.12	−5.88	0.02582	1.59	0.0460
4	13.97	−5.88	0.01033	7.97	0.2305
3	14.34	−5.88	0.00513	0.36	0.0104
	Pooled values	Mean square	0.0093		0.1416
		Standard deviation	0.096		0.376
Hart Samples, E-Scale Readings, 70°F, Sorption, Special Preparation					
4	8.33	−6.39	0.02541	0.5904	0.0145
3	9.86	−6.39	0.09023	0.3770	0.0092
4	12.68	−6.39	0.00895	0.5666	0.0139
3	13.34	−6.39	0.01489	1.9949	0.0489
	Pooled values	Mean square	0.0313		0.0201
		Standard deviation	0.177		0.142
Steinlite samples, B-Scale, 3.0-Inch Stop, 70°F, Desorption, Standard Preparation					
6	8.57	0.2238	0.01951	0.00102	0.0204
4	9.88	0.2238	0.00000	0.00015	0.0030
4	13.97	0.2238	0.03479	0.00086	0.0172
4	14.30	0.2238	*	0.00000	0.0000
	Pooled values	Mean square	0.0184		0.0116
		Standard deviation	0.136		0.108
Steinlite Samples, C-Scale, 3.75-Inch Stop, 70°F, Desorption, Standard Preparation					
3	5.52	0.0678	0.06510	0.0000637	0.0139
3	11.03	0.0678	0.01873	0.0000225	0.0049
	Pooled values	Mean square	0.0419		0.0094
		Standard deviation	0.205		0.097
Steinlite Samples, C-Scale, 4.50-Inch Stop, 70°F, Desorption, Standard Preparation					
3	5.52	0.1503	0.06713	0.0006310	0.0279
3	11.03	0.1194	0.02110	0.0001555	0.0109
2	14.06	0.1025	0.02706	0.0002917	0.0278
	Pooled values	Mean square	0.0407		0.0211
		Standard deviation	0.202		0.145
Pooled standard deviation for all Steinlite samples 0.115					

* Irregular and not included in analysis.

converted to logarithms; therefore, in this instance, Y refers to the logarithm of the meter reading.

Residual Y variances calculated from the square of deviations of Y upon X may be considered as a measure of the over-all range of expected instrument readings for any given value of X. However, the variances so calculated include those for samples as well as those for the instrument, and are not a pure evaluation of what may be expected from the instrument alone. While it is difficult to separate these two effects, particularly with bulk samples, an estimation is presented below, which would appear to be reasonably reliable as indicated by the consistency of the results.

Variance Due to Instruments

If sheet fabrics were being tested, measurements in the directions of the warp and the fill, with an analysis of variance to indicate the residual error not attributable to sampling, would be relatively straightforward. With bulk materials, the procedure is not so simple, particularly since some instruments require a large amount of sample and others a relatively small amount.

In subsequent discussion, instrument and sampling errors will be based upon the groups of points obtained when materials were similarly conditioned. The resulting estimates of variances will be pooled to give an over-all estimate of variance for the instrument (or oven). It will be assumed that between groups the variances are homogeneous and that in any group of points the variance on either the X or the Y-scale is a summation of the effects of the sample and the method. Therefore, the estimate of X-variance, V_X, and the estimate of Y-variance, V_Y, will be written as follows:

$$V_X = V_S + V_O; \quad (1)$$

$$V_Y = \beta^2 V_S + V_I; \quad (2)$$

where V_O refers to variance introduced by oven test method only, V_I refers to variance introduced by instrument test method only, and V_S refers to variance introduced by samples only.

In equation (2), β is the slope of the general Y vs. X line at the average X value for the group of points considered. It will be constant for the entire calibration line only if a second-order dependence of Y upon X is not involved. Since the sample variance should appear on both X and Y scales, its value on the X-scale should be multiplied by β^2 to indicate its contribution on the Y-scale. In addition to the relations already written, it may be concluded that the co-variance, V_{XY}, should not include the instrument and oven variance; thus, it should be legitimate to write

$$V_{XY} = \beta V_S. \quad (3)$$

Substituting equation (3) in equations (1) and (2), the oven and instrument variances may be expressed in terms of the X-, Y-, and XY-variances as follows:

$$V_O = V_X - 1/\beta \; V_{XY}; \quad (4)$$

$$V_I = V_Y - \beta \; V_{XY}. \quad (5)$$

Results of calculations from these equations are noted in Table I.

The variances so calculated are, of course, estimates based upon the sample covered by experiment and are therefore more properly termed "mean squares." They may be pooled for the individual groups of points along a calibration line in a standard manner. Estimates, s, of the standard deviation, σ, are obtained as the square roots of the V terms, and two-s limits should give an estimate of the ranges in which 95 percent of all points should lie based upon the usual theory of normal distribution. These ranges on the Y-axis must be reconverted through the general slope of the line to give a comparative range on the X or moisture scale.

Results

Instruments

For the instrument variance with the Steinlite, V_I values from equation (5) show no marked effects in the different density ranges. Pooling of all values seems the most desirable procedure, the estimate being 0.115 (Table I). There would, therefore, be no indication that any particular density is preferred for precision. It is apparent, however, that very low moisture values on a curve whose character is concave upward will eventually reach a flatness such that reduction in precision is unavoidable. The effect may be readily shown by parallel two-sigma lines on the highest-density, C-scale calibration line. (This line was obtained by converting B-scale readings, not by direct measurement; the two-sigma limit lines are obtained in the same manner.) In the concave downward curves, the upper end does not become sufficiently flat to cause this trouble.

When converted through the slope of the regression line, the two-s values for the Steinlite instrument indicate that 95 percent of all experimental points should fall in a range of approximately 0.5 percent moisture (Table I). The same type of calculation for the Hart instrument indicates an equivalent range in the case of one type of sample, but a much higher one, 1.5 percent, in the case of another. This last relatively high range is interpreted as indicating the existence of another variance inadvertently included in the sample because of the type of electrode. As discussed in the main body of this paper, the Hart reading is dependent upon the thickness of the sample, and although every attempt was made to maintain uniform thickness of the samples, it would appear from the variances just calculated that these attempts at uniformity were not suc-

TABLE II. POOLING OVEN "VARIANCES" BASED ON GROUP MEANS

n = number of samples
k = number of duplicates in a sample

Meter test	k	V_o	$n-1$	Sum of squares $\Sigma\ V_o(k)(n-1)$	Σ (Degrees of freedom)
Hart standard	3	0.00000	3		
	2	0.02582	2		
	3	0.01033	3		
	2	0.00513	2		
				0.21677	10
Hart special	3	0.02541	3		
	2	0.09023	2		
	3	0.00895	3		
	2	0.01489	2		
				0.73800	10
Steinlite, B, 3-in.	3	0.01951	5		
	3	0.00000	3		
	3	0.03479	3		
				0.60576	11
Steinlite, C, 3.75-in.	3	0.06510	2		
	3	0.01873	2		
				0.50298	4
Steinlite, C, 4.50-in.	3	0.06713	2		
	3	0.02110	2		
	3	0.02706	1		
				0.61056	5
			Total	2.67407	40

$$\text{Pooled variance} = \frac{2.67407}{40} = 0.0669$$

Pooled s = 0.259

cessful in the case of the standard samples. The fact that the instrument gave the same range of reproducibility as the Steinlite on one set of samples indicates that it is possible to obtain this precision, but the difficulty of maintaining it is indicated by the lack of consistency between sample types. This problem should be less acute when the Hart is used for sheet materials of uniform thickness.

Oven Samples

For both meters, samples were taken from skeins in an identical manner. Therefore, oven variances from both the Hart and Steinlite should be of the same order. Reasonable duplication was obtained and is considered to be evidence in favor of the estimation method used.

Equation (4) above has been used to eliminate sample variance from oven variance, with results as reported in Table I. However, another method of effecting the same separation is available in a standard analysis of variance, since duplicates of the oven moisture were obtained for each sample at any condition. Each point on the calibration line represents an average of several oven determinations which may be considered as duplicates, and each point represents a different sample. Thus a standard analysis-of-variance table can be established with dimensions of samples and duplicates.

In comparing "within-sample variances" from this analysis-of-variance table with oven variances obtained from equation (4), it must be borne in mind that the latter values are calculated upon sample means with the former on individuals. Therefore, any comparison of the two must be made by conversion to the same basis. The V_o values have been chosen for conversion and have been handled in the following manner. Each value has been multiplied by the number of sample duplicates giving a variance based upon the individuals. This procedure is quite straightforward but the pooling of these vari-

TABLE III. POOLING OF OVEN "VARIANCES" BASED ON "WITHIN-SAMPLE" ESTIMATES

Source	Degrees of freedom	$s^2(n-1)$	Sum	Pooled s_p
Hart standard	3	0.0975		
	3	0.0145		
	8	0.4726		
	8	0.1813		
			0.7659	0.186
Hart special	3	0.1385		
	3	0.7028		
	8	0.7825		
	8	0.1372		
			1.7610	0.282
Steinlite, B, 3-in.	8	0.1323		
	8	0.1813		
	12	0.4389		
	8	0.3824		
			1.1349	0.178
Steinlite, C, 3.75-in. and 4.50-in.	6	0.2135		
	4	0.0797*		
	6	0.1141		
			0.4073	0.160
Total	96		4.0691	
			Pooled	0.206

* 4.50-in. stop only.

ances is not so simple. The most reasonable procedure seems to be consideration of the number of degrees of freedom involved as equal to the number of samples minus one. The variances of the individuals are therefore multiplied by this number of degrees of freedom, the sum of squares so obtained is added, and the total is divided by the sum of the degrees of freedom for all tests. This procedure is indicated in Tables II and III.

From the pooled estimate of variance by equation (3), a standard deviation of 0.259 was calculated. The comparable figure from the analysis-of-variance table is 0.206. These figures are in close enough agreement to confirm the adaptability of the methods chosen for analysis of the data.

Conclusions

It will be further noted that the estimates of oven variability are larger than, but of the same order of magnitude as, the best of those obtained with the instruments when the latter are converted to the moisture scale, and it is therefore possible to conclude that the instruments are capable of reproducing their readings at least as closely as the ovens may reproduce theirs. The actual magnitude of the estimates of the standard deviations is unquestionably dependent upon the method of test used as described in the main body of this paper. But, since all the results are calculated on samples conditioned to or near equilibrium, it is probable that the precision indicated by these calculations is a fair estimate of what may normally be expected in ordinary use.

Statistics for Grab Samples

If samples are taken by the grab-sample method rather than from samples conditioned to equilibrium, an additional time variance would be added to the terms in equations (1) and (2). It would not, however, be proper to assume that this variance is uniform, nor would it be possible to conclude that it would be similarly imposed on both the X- and Y-scales. Thus, there is no relation by which it can be removed from both scales as in the previous analysis. While it may be concluded that statistical analysis of grab-sample calibrations is impossible, the conclusion that grab samples have no usefulness in calibration does not follow. If reasonable care is exercised, as described in this paper, grab-sample runs may be used to fill in the space between equilibrium points or even to determine the shape of the entire calibration line. Variances calculated from such lines will not be necessary if the material in this paper is accepted as a guide.

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A Method for the Electron Microscopy of Wool*

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Abstract

A simplified method for making shadowed thermoplastic negative replicas of fibrous materials was developed. Through this technique fine details of the morphology of the wool fiber, before and after treatment, were clearly revealed by the electron microscope. The structural implications of the electron micrographs are discussed with respect to the subject of wear and with reference to the observations of other investigators.

I. Introduction

1. The Problem

The electron microscope has been a useful tool for observing objects which are thin enough or which are sufficiently small and adequately dispersed to allow the proper scattering and transmission of electrons in the conventional 50 KV microscope.‡ The high resolving and magnifying powers of the electron microscope have hence been used with considerable success in the study of pigments, smokes, viruses, bacteria, and a variety of other colloidal particles.

On the other hand, when efforts are made to study organic fibers such as wool, ordinary techniques of direct specimen preparation fail. In the first place, the fiber is too thick to transmit a sufficient intensity of electrons to excite the phosphor-coated screen into luminescence or to form an image on the photographic emulsion. At best, only a profile view can be obtained when a section of the fiber is inserted in the specimen holder. Second, and no less troublesome, is the fact that in scattering most of the electron beam there is an exchange of energy. The subsequent generation of heat and electrostatic charges causes a charring and disintegration of the fiber itself. Third, the necessity of operating the electron microscope at a pressure of less than 10^{-4} mm. of mercury results in desiccation of the specimen. The literature of the past few years is replete with descriptions of limitations which make observation and interpretation difficult for the electron microscopist.

2. Evaluation of Existing Techniques

Partial solutions to the problem have been found by those concerned with the study of wool fibers. Fragments of triturated fibers of reduced and methylated wool are not adversely affected by the intense beam of electrons and are, in part, transparent to it. Such techniques, however, are very limited. Not only do they preclude getting detailed information about the surface structure of the fiber, but they also eliminate the possibility of observing the same fiber before and after treatment because of the mechanical disorganization and chemical alteration which these techniques produce [2–4, 7, 10, 11].

The limitations of direct methods of surface observation by electron-optical means have led to the development of ingenious and diverse techniques for preparing thin film replicas of the surfaces of electron optically "opaque" objects [12, 17]. The improvements and variations of the basic replica methods reported by a number of resourceful workers have been successfully applied to the study of many bulk materials [1, 6, 9]. None of these techniques, however, is easily applicable to the study of degraded wool fibers, since the use of excessive heat or crushing pressure in molding the replica introduces undesirable changes. Interpretation of results is often complicated by artifacts. Moreover, most replica methods involve destruction of the specimen. This, too, eliminates the possibility of performing additional treatments on the same fiber.

In order, therefore, to augment the data on the structure of the wool fiber and to correlate these

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† Now with the Office of Naval Research.

‡ The RCA Type EMU electron microscope was used for this work.

observations with results previously obtained by the direct disintegration of the fiber as well as by other methods, a simple, rapid, faithful replica technique was developed. This system of specimen preparation successfully combines methods useful for both optical and electron microscopy [5]. A negative replica of the wool fiber is made in a thermoplastic film which is thin enough to examine directly in the electron microscope and which may be shadow-cast with a suitable metal for added contrast.

II. Replica Method

1. Materials and Apparatus

Replicas can be made with two thermoplastic materials. One, ethyl cellulose,* is dissolved in ethyl acetate. Addition of about 10 percent of ethyl alcohol aids solution. The other thermoplastic which yields a slightly stronger film and one which withstands electron bombardment better is polystyrene.† Styron granules dissolve in benzene. Warming the solvent slightly hastens the reaction. Solutions of 1 to 2 percent (by weight) seem to be satisfactory for each resin. The principal criterion is the ability of the plastic films to transmit a sufficiently intense beam in the electron microscope. The films formed by evaporation of the solution after it is poured onto standard, glass, microscope slides are thicker than the substrate films usually employed for electron microscopy. About ¾ of the slide is coated with the thermoplastic resin. The excess is drained off into a towel by placing the glass slide in an almost vertical position. After 5 minutes of drying a slightly wedge-shaped film, averaging about 0.4 microns in thickness, is formed.

The fibers to be studied should be rinsed in ethyl alcohol and/or ether to remove surface grease and dirt.‡ After being dried at room temperature these fibers are then singly placed upon the thermoplastic lacquer which is on the glass slide. Another glass microscope slide is then placed on top of the fibers, sandwiching them with the plastic film. This sandwich can be firmly held together with the aid of a C-clamp and a couple of strips of cardboard and heat-resistant rubber cut to fit the glass slides. Reasonable care must be exercised not to twist the assembly out of alignment. The rubber and cardboard pads take up most of the torque and contact pressure produced when the clamp screw is tightened, by hand, to the point where the sandwich will be firmly fixed without breaking the glass and without displacing the arrangement. The clamped glass sandwich of film and fibers is then heated in a laboratory resistance-type oven for about 20 minutes at 90°C. The film is thus softened to the point where it will flow and accept the wool fiber and form a faithful replica of the surfaces in contact with it.

The variable factors that affect the quality and resolution of the plastic replica are time, temperature, pressure, thickness of the film, and diameter of the fiber. If the thermoplastic is allowed to remain in the oven for too long a time or at too high a temperature, too much plastic flow and deformation will result. If too much pressure is applied, the displaced material will pile up on each side of the fiber and will disturb the quality and appearance of the replica (Figures 8 and 9). If the film is too thin for the diameter of the fiber, the fiber will be pushed through it to the glass and will prevent the plastic from molding itself about the fiber surface. To achieve the proper balance of all these elements is not a difficult problem inasmuch as the limits of control are not critical. One or two trials ordinarily provide sufficient experience so that numerous acceptable replicas can be made in a comparatively short time. After the clamped sandwich is removed from the oven and allowed to cool to room temperature it can be separated. Usually the wool fibers come away with the uncoated glass slide, leaving semicylindrical impressions in the plastic. If not, it is a simple matter to remove them whole with the aid of a fine brush or tweezers. These fibers having once been accurately molded can be saved for further chemical or physical treatment and subsequent replication.

2. Metallic Shadow-Casting

Metallic shadow-casting adds contrast as well as a three-dimensional aspect to the surface details of the replica [15, 16]. A very thin coating of evaporated chromium is deposited obliquely upon the plastic surface. The elevations and depressions of the surface replica cast characteristic shielded radiation shadows. Where no metal is condensed, transmission of the incident energy is a maximum. Where thick elevations and ridges occur coupled with a layer of metal, electron scattering is a maximum and little or no energy is transmitted.

* Sold commercially as Faxfilm, Ethocel, Ethofoil, etc.

† Styron (NP 6—K27) made by Dow Chemical Co.

‡ The wool used for this work had been commercially degreased. Prolonged washing was, therefore, unnecessary.

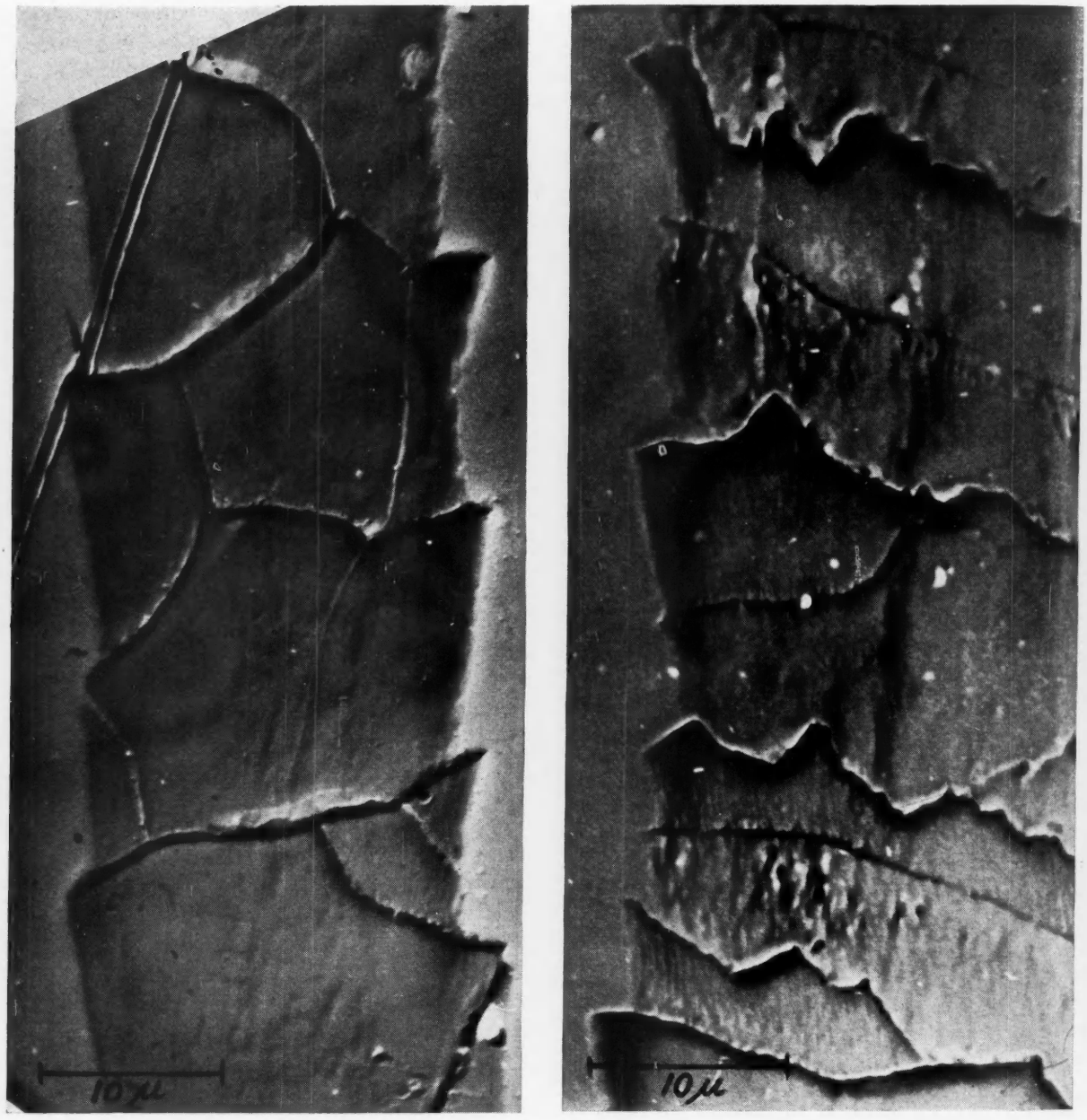

FIG. 1 (left). Tot. Mag. ⌣ 2700 × (el. 716 ×, opt. 3.8 ×)

Typical chromium-shadowed polystryrene negative replica of a wool fiber taken from unabraded, undyed, fine worsted fabric. The normal appearance shows the smooth lacquer-like coating on the imbricated cuticle. Faint striations may be seen which are part of the corrugated scale structure lying below this amorphous outer layer.

FIG. 2 (right). Tot. Mag. ⌣ 2900 × (el. 716 ×, opt. 4 ×)

Chromium-shadowed polystyrene negative replica of a wool fiber taken from a mechanically abraded fine worsted fabric. The slight amount of abrasion removed the lacquer-like outerlayer of alternate areas. The middle area was protected from wear by the interlaced nature of the fabric.

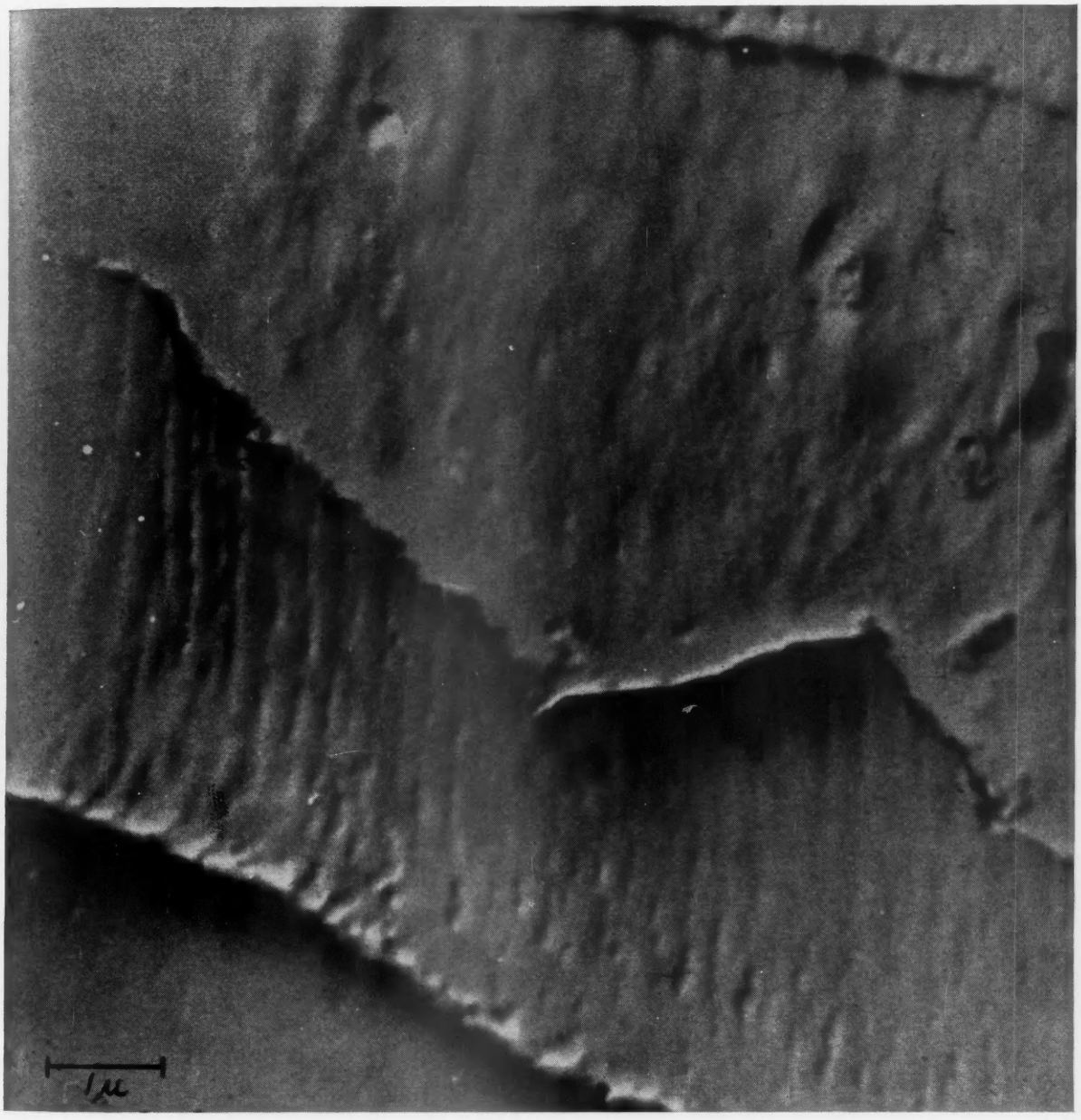

FIG. 3. Tot. Mag. ∽ 16,500 × (el. 3600 ×, opt. 4.6 ×)

An enlargement of the abraded area in the lower part of Figure 2 showing the corrugated or ribbed underlying structure of the cuticle, in contrast with the smooth surface of the unabraded fiber (lower left). The longitudinal striations are an integral part of the scales themselves and not a reflection of the cortical-cell formation underneath. Note the overhanging portion of a scale, in the upper left area of the replica, under which there was no cortex.

FIG. 4 (left). Tot. Mag. ⌣ 2900 × (el. 716 ×, opt. 4 ×)

Chromium-shadowed polystyrene negative replica of a wool fiber taken from the "debris" of mechanically abraded fine worsted fabric. Except for a few remnants, the scales have been removed and another comparatively smooth layer of material, the intercellular cement, lying between the cuticle and the cortex is exposed.

FIG. 5 (right). Tot. Mag. ⌣ 2400 × (el. 716 ×, opt. 3.4 ×)

Chromium-shadowed polystyrene negative replica of a wool fiber taken from the debris of mechanically abraded fine worsted fabric. Some scale remnants may be seen, but for the most part the coarse striated structure is that of the cortical cells beneath, indicating a greater degree of abrasion than that shown in Figure 4.

FIG. 6. Tot. Mag. ⌐ 3300 × (el. 716 ×, opt. 4.6 ×)

Chromium-shadowed polystyrene negative replica of the remains of a wool fiber from severely mechanically abraded worsted fabric. The size of the structure lying across the center is about that of a cortical cell. Inasmuch as the soft cortex is easily deformed and the fiber is in the last stages of its filamentary formation, interpretation is questionable.

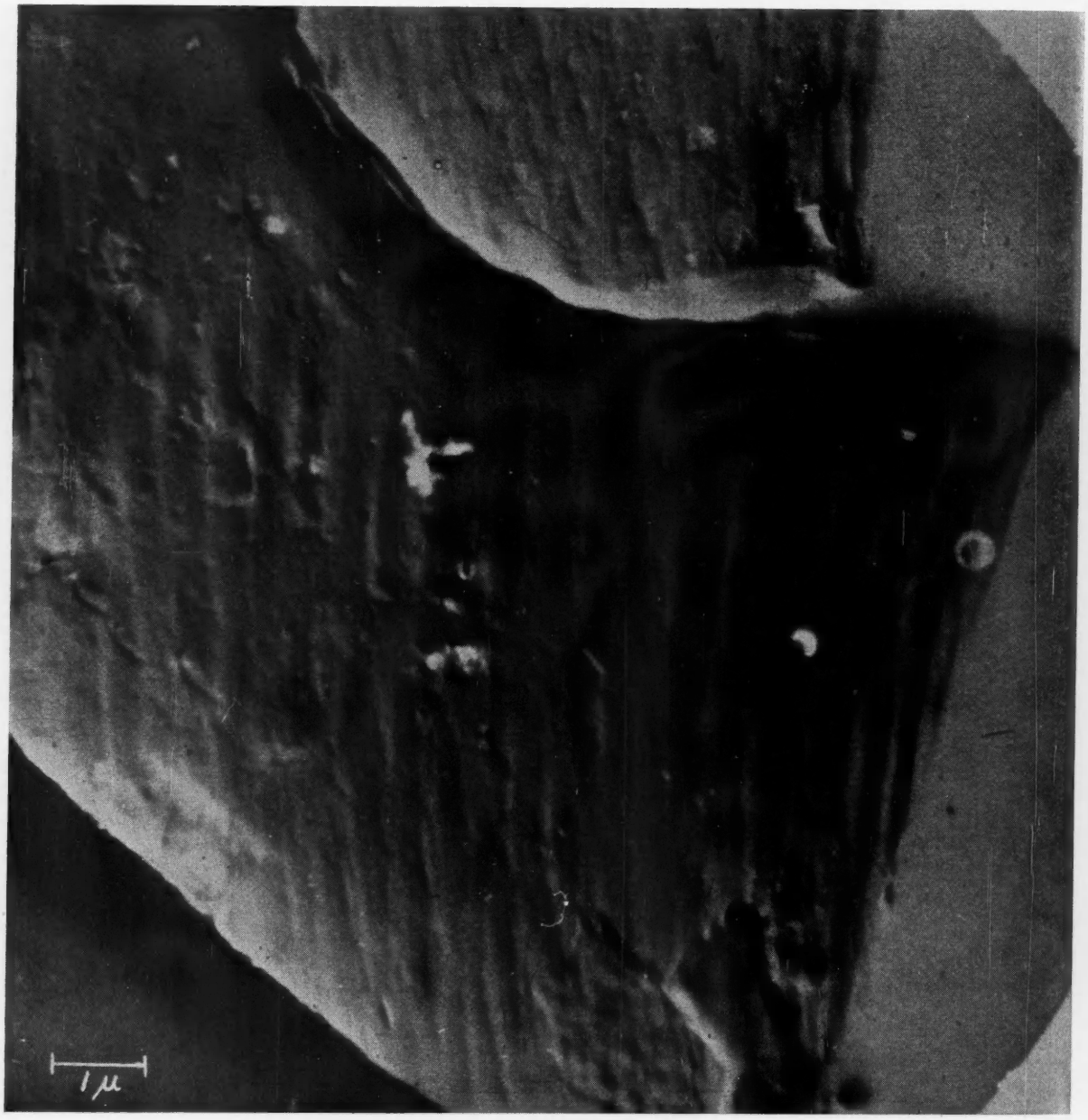

FIG. 7. Tot. Mag. ∽ 13,300 × (el. 3600 ×, opt. 3.7 ×)

Chromium-shadowed ethyl cellulose negative replica of an atypical fiber taken from unwashed, undyed, worsted yarn showing slight surface degradation associated with the previous history of the wool. Note the undegraded, smooth scale surface in the lower left corner. Here the thicker lacquer-covered scale is more deeply imbedded in the thermoplastic film.

FIG. 8. Tot. Mag. ~ 2600 × (el. 540 ×, opt. 4.8 ×)

Chromium-shadowed ethyl cellulose negative replica of two fibers from an unwashed, undyed, worsted yarn showing the bulging distortion of one resulting from excessive contact pressure on the fiber. Note that the scale structure has stretched to more than double its surface area in order to accommodate the deformation without rupture. The smooth outerlayer is still intact, indicating its plastic nature.

FIG. 9. Tot. Mag. ⁓ 3600 × (el. 716 ×, opt. 5 ×)

Chromium-shadowed polystyrene negative replica of wool fibers taken from untreated khaki-dyed fabric. The normal surface structure of one fiber may be compared with the features of one damaged by an unknown agent, probably chemical.

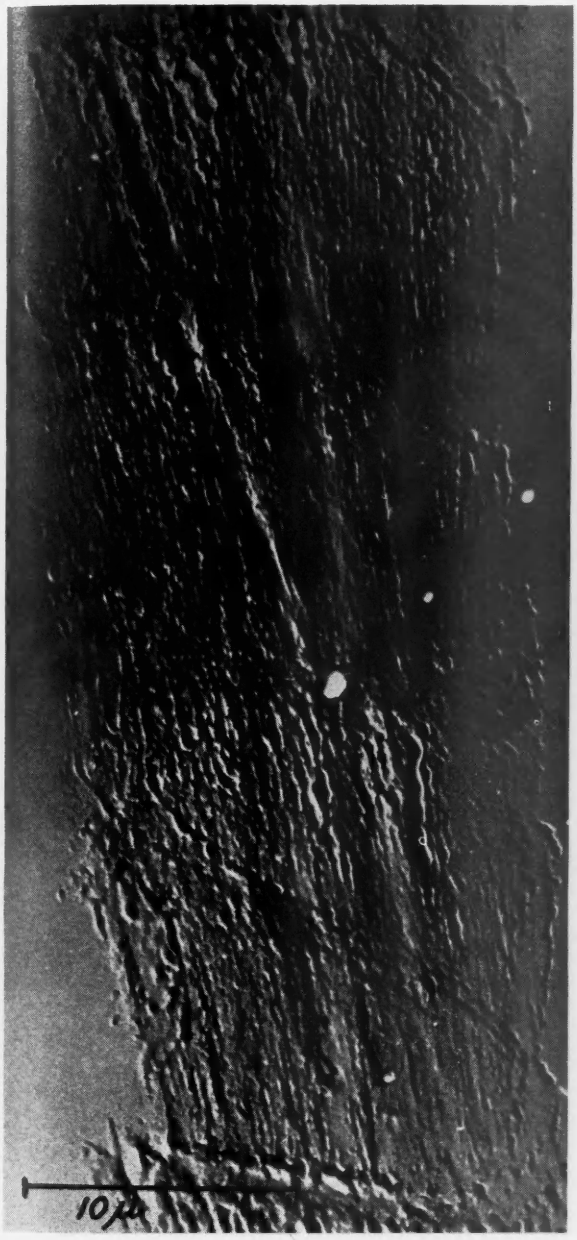

FIG. 10 (left). Tot. Mag. — 3600 × (el. 716 ×, opt. 5 ×)

Chromium-shadowed polystyrene negative replica of a fiber taken from undyed worsted fabric, immersed in a 1% aqueous solution of calcium hypochlorite long enough to wet it, and then rinsed in cold water. The fiber still exhibits its filamentous structure.

FIG. 11 (right). Tot. Mag. — 17,000 × (el. 3600 ×, opt. 4.7 ×)

An enlargement of the area in the lower part of Figure 10. The erosion of the superficial lacquer-like coating exposed the characteristic striated substructure of the scales which was more resistant to the chemical attack.

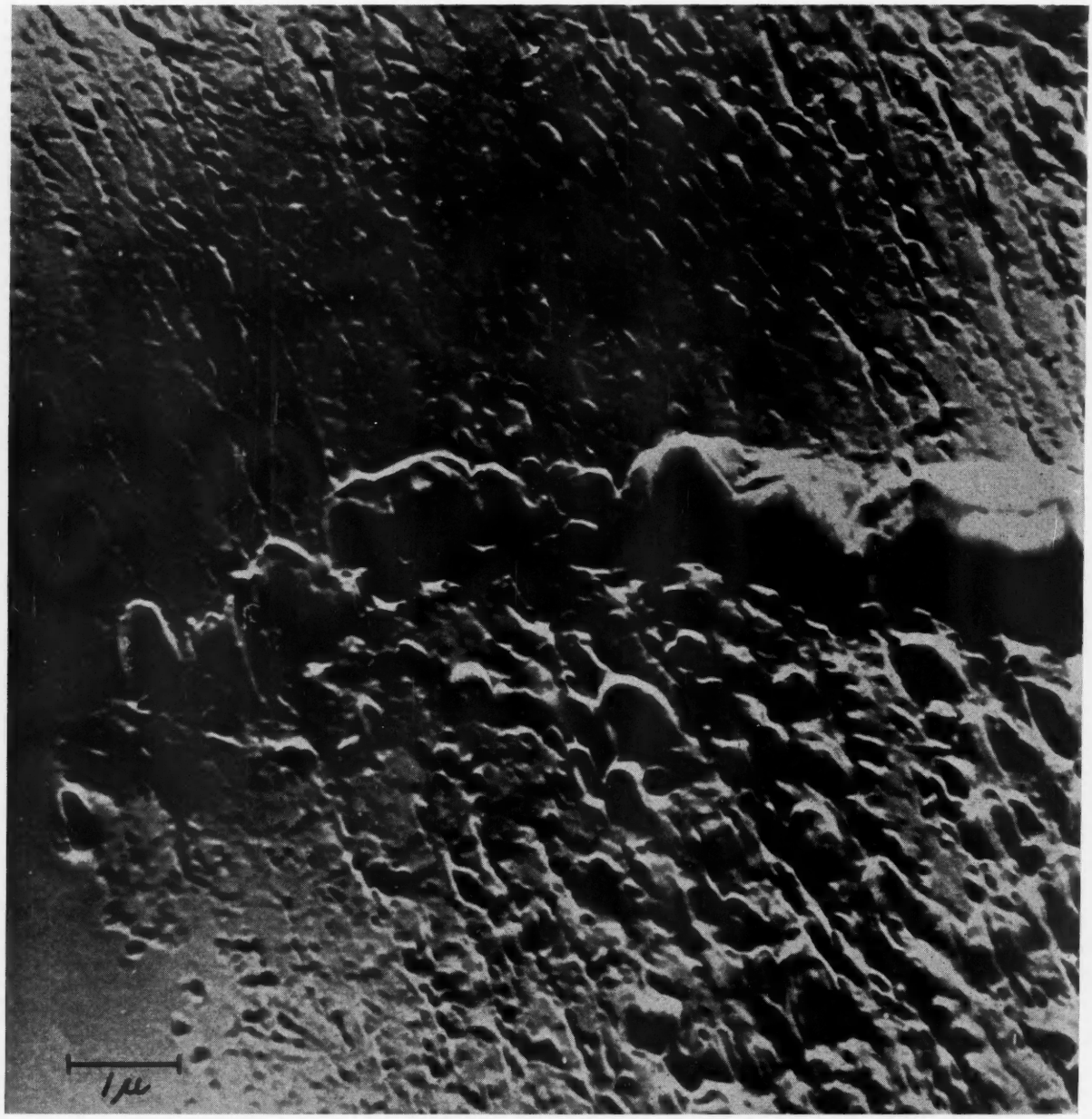

FIG. 12. Tot. Mag. ∽ 16,500 × (el. 3600 ×, opt. 4.6 ×)

Chromium-shadowed polystyrene negative replica of a wool fiber after chlorination, showing what is probably the blistery sacs of the Allwörden reaction on the lacquer-like scale surface, with evidence of concentrated attack at the edges of the scale.

The glass slide bearing the plastic film containing the replicas of the fibers is placed horizontally with the film side up in a vacuum chamber where it can be shadowed.* To prevent any appreciable heating, the thermoplastic replicas are placed a distance of 25 cm. away from a conical basket made of 30-mil tungsten wire. The basket may be formed by winding the tungsten around the pointed end of a No. 10 wood screw. Heating the wire and screw helps to prevent brittle fracture during the winding process. If instead of the 30-mil wire a twisted double strand of 16- or 20-mil tungsten wire is used, no heat is required, and it is easier to follow the troughs in the screw thread. The additional surface area provided by the stranded wire is advantageous. A 6-inch length of wire is sufficient for a basket about a centimeter high, with leads of adequate length. The tungsten basket is set in the vacuum chamber so that the apex of the cone points downward and is 5 cm. above the plane of the replica. A charge of 100 to 150 mg. of pure chromium † metal fragments is placed in the basket. All of the metal is evaporated slowly with a current of 25 to 35 amperes at 10 to 15 volts in about 10 minutes at a pressure of 10^{-5} mm. of mercury. A 10-minute outgassing period with 10 to 20 amperes running through the tungsten wire is a desirable practice.

The uniformity of deposition of the metal as it condenses on the specimen is a function of the mean free path and the accommodation coefficient. The latter depends upon the initial temperature, the emission temperature, and the striking temperature of the molecules or atoms of the metal. Given a large accommodation coefficient and a vacuum where the mean free path is about a meter in length, the molecules will traverse the distance from filament to specimen without collision and impinge upon the comparatively cold surface of the specimen with very high energy. If these conditions are not met, the molecules of metal will not stick where they land but will migrate, find other molecules, and form undesirable agglomerations of recrystallized metal. This produces a grainy, irregular deposition. At high magnification the poorly defined shadowed areas and the graininess of the surface provide undesirable effects.

* The RCA EMV unit was used for this work.

† Electrolytically deposited chromium about 1/16 in. thick was used.

3. Selection and Examination

The chromium-shadowed thermoplastic negative replica, still on the glass slide, is viewed in an ordinary light microscope. Photomicrographs may now be taken, in the manner of Hardy and Plitt [5]. The replica may also be projected in a photoenlarger, in the manner of Scott and Wyckoff [14]. Or, desired areas may be selected and mounted for more detailed observation in the electron microscope. In the latter case, the woven, calendered, 1/8-inch stainless steel, No. 200 U. S. Standard Sieve specimen screens are centered over the chosen areas of the replica, with the aid of a low-power light microscope. The screens usually adhere to the chromium coating, especially if contact is made with the convex side. No. 100- or 150-mesh screens would offer larger areas for observation. The glass slide bearing the replica film and the specimen screens is slowly immersed at almost grazing incidence into a 9-inch crystallizing dish of fresh tap water. The thermoplastic film can be readily floated off the glass slide with little or no teasing, especially if the edges of the glass slide where the plastic may have overrun are rubbed with the side of a dissecting needle or similar tool and no water is allowed to creep up over the film. The freely floating film supporting the specimen screens is removed from the water by flipping it over and out with a piece of paper about the size of a glass slide.

The screens are thereby lodged between the film and the paper. Any short-fibered paper may be used for this purpose. The short fibers prevent wrinkling and warping upon drying. Unprinted newspaper, porous filter paper, melamine resin-bonded map paper, blotting paper, or the strips used to separate the glass in a box of microscope slides may be used with about equal success. When paper is used instead of glass to retrieve the floating film, the specimen screens supporting the selected areas of the replica can be readily removed after drying. To facilitate this, however, it is advisable to prick the film about the periphery of the screen with a needle point while the film and paper are still damp. A faster and easier technique, which, however, involves some risk, is accomplished by spreading a small amount of a suitable solvent on glass and placing the dried paper in contact with it. The solvent soaks through the paper and up to the plastic film. When the solvent evaporates, the paper is dried flat to the

glass, the plastic is cemented to the paper, and the untouched areas of the replica supported by the specimen screens can be picked up with tweezers as easily as from a much thinner film. Obviously, if too much solvent is used the entire replica may be ruined. If a reasonable amount of caution is observed, however, this catastrophe will not occur. This technique provides a much more advantageous procedure for removing the specimen than any method of cutting or tearing the comparatively thick film from a hard glass slide or a coarse wire gauze.

Various workers in the field have suggested that a possible improvement in the technique for selecting and mounting the replica film on a specimen screen could be made by placing the film, replica side up, over a piece of 100-mesh wire cloth. After scanning the film for suitable areas with a light microscope, an auxiliary mount in the revolving nosepiece fitted with a cutting die could be turned into place and the tube racked down. In this manner, the desired area of film may be punched out with a supporting screen attached. The adaptation of a microscope-punch combination seems worth while not only for this work but in many other aspects of electron microscopy where systematic scanning is required for selecting areas of a prepared specimen.

III. Application and Interpretation

The shadowed replica method was applied to the study of wool fibers as reflected by the characteristic changes after varying amounts of abrasion produced by an apparatus designed by Schiefer [13]. During the abrasion cycle fiber fragments fall from the abradant. This "debris," as well as the fibers bordering the hole worn through the woolen fabric, was examined. Hundreds of replicas were made of fibers abraded in this way. For comparison, replicas were made of untreated fibers, khaki-dyed fibers, as well as fibers subjected to chemical attack. The shadowed replicas were examined in the electron microscope at low magnifications obtained without the projector polepiece, as well as at the higher ranges of magnification. Electron micrographs were made with a 1-mil objective aperture and with 5–10 seconds' exposure. Contact photographic reversals were made on another medium lantern-slide plate for further optical enlargement. Negative prints are shown in the figures. For proper interpretation of the surface features of the negative replicas, they may be viewed as though they were obliquely illuminated. The "diameter" indicated in the micrographs represents 80–90 percent of the actual diameter of the wool fiber.

A wool fiber when plucked from the living animal consists of two distinct regions. The root is the living part and grows from the hair follicle. The nonliving portion that emerges from the root is called the shaft, which is the section shorn from the sheep and is here designated the wool fiber [8]. It generally consists of three principal concentric layers—a thin outer covering of scales (cuticle), a middle region (cortex), and a central core or pith (medulla). The relative thickness of each layer varies from fiber to fiber as well as from sheep to sheep. In the very fine wool fibers, the medulla is often absent and a few comparatively large scales encircle the cortex. The spindle-shaped cortical cells comprise the main bulk of the fiber. It is well known that the ability of wool fibers to felt, tangle, and curl is dependent upon the direction and shape of the imbricated scale structure and that this cuticle serves to protect the fiber from chemical and mechanical degradation (Figures 1 and 8). It has been recently suggested by Mercer *et al.* [3, 10, 11] that these three dominant cellular structures are encased in an amorphous material which functions as a matrix and as an intercellular cement and that the cuticle layer may be covered by this same featureless keratinous material (Figures 1–6). These investigators and others have shown this amorphous plastic outer covering of the scales to be readily digested in an enzyme, as is the featureless intercellular cement which exists between the scale and the cortex, as well as between and within the cortical cells themselves. The similar reaction to enzymic treatment suggests similar composition. Microscopy alone cannot disclose whether there exists a continuous matrix of a single substance which envelopes the differentiated cell structures or whether the keratinous material is made up of different protein molecules with varying properties.

The present study confirms, at least partially, the "matrix theory" of the morphology of the wool fiber. Electron micrographs of the surface replicas of mechanically abraded and unabraded wool fibers (shown in Figures 1, 2, and 3) reveal that the scale is heterogeneous in structure and consists, at least, of a double layer. The outer layer is a smooth covering for the under structure and shows no specific surface features (Figure 1). When this lacquer-like coating is removed by mechanical abrasion, a rigid striated

structure is exposed (Figure 2). These regular corrugations (ridges and furrows) run more or less parallel to the longitudinal axis of the fiber. They are a feature of the scale itself and are not dependent upon, or a reflection of, the size and formation of the cortical cells. This is borne out by the fact that the ribbed structure is seen in the overhanging portions of the scales under which there is no cortex (Figure 3). With greater degrees of abrasion, the scales are polished away, leaving a smooth filamentary structure with slight vestiges of the former imbricated surface (Figure 4). Still more rubbing removes the next amorphous layer under the scales and reveals the cortical cells themselves imbedded in the cementum (Figure 5). After such severe abrasion, the wool no longer manifests its characteristic filamentary structure and, as such, is destroyed (Figure 6).

Thus far, the present investigation indicates that the weaving process introduces very little change in the surface structure of the fibers. Some portions show degradation of the scale, but this is by no means typical. When compared with unwoven, undyed fibers, less than 10 percent of the fibers studied showed any modification (Figure 7). These alterations are very likely associated with the previous history of the fiber. They are not necessarily a result of the weaving process. In the case of khaki-dyed woolens, similar evidence was noted (Figure 9). In a few samples where the fibers were not washed in alcohol, some crystalline material was found imbedded in the replica. This may have been some of the dyestuff which had lodged on the outer scale surface. In the case of fibers which had been chlorinated in a 1-percent aqueous solution of calcium hypochlorite long enough to wet the entire length and rinsed in a large quantity of cold water (Figures 10, 11, 12), the sacs and blisters formed (probably the Allwörden reaction) indicated a violent attack on the amorphous lacquer-like layer of the scales. The general erosion which took place exposed the characteristic striated substructure of the scale which withstood the action of chlorine. The exposed scale edges showed the greatest amount of attack (Figure 12). The treatment given the fibers was, of course, much more drastic than that used in some commercial processes for shrinkproofing woolens.

With the foregoing method, the structural changes in textile fabrics resulting from various physical and chemical factors can be studied. Alterations in the physical structure are brought about by such means as abrasion, flexion, tension, acid, alkali, chlorine, heat, sunlight, and other elements of degradation. Wear is a complex phenomenon with a large variety of contributing factors. Before statistical correlation of any microscopic observations of laboratory wear versus actual wear may be obtained, a large number of tests interpreted against a broad background of performance in service is necessary. The "polishing" produced with Schiefer's uniform abrasion machine offers a systematic approach to a microscopic study of a worn fabric. The characteristics of fiber structure may in turn be correlated with the morphological modifications resulting from actual wear in service. While the fundamental problem of the molecular structure of organic fibers cannot be solved by electron microscopy alone, considerable information may be gained that is of interest to the textile technologist and to those concerned with the subject of wear-resistance.

Acknowledgment

The authors take pleasure in acknowledging the very valuable assistance of Esther Golovato, in processing the many photographs, and H. F. McMurdie and W. D. Appel, without whose able direction much of this work would not have been done.

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(Manuscript received June 24, 1948.)

Fundamental vs. Basic Research

"Fundamental research—pure scientific investigation undertaken for the love of knowledge and without any eye on its potential usefulness.

"Basic research—similar to fundamental research, but embarked on with one eye on industrial application.

"Development—the link between basic work and actual production."

These definitions were presented by R. K. Sanders in a commentary regarding research in "Research" (London).

Would it not assist in clarifying our understanding of the functions of research were these distinctions to be generally adopted?—EDITOR.

The Partial Carboxymethylation of Cotton to Obtain Swellable Fibers, II*

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Southern Regional Research Laboratory,† New Orleans, Louisiana

THE AUTHORS have previously described [7] their work on the partial carboxymethylation of cotton linters, yarns, and threads for the purpose of obtaining quickly swellable fibers to aid in producing a cotton cloth less permeable to water. The present paper describes work on carboxymethylation in which cotton cloth was treated in the piece and cotton yarn was treated before it was woven into cloth. Whereas the cloth treated directly showed some improvement, the cloth woven from the treated yarn would not swell sufficiently when wet to close all interstices and prevent water leakage. The cloth from the treated yarn was, however, highly absorbent.

A similar product, carboxyethylated rayon, prepared by the action of acrylonitrile on viscose, has recently been described [4].

Treatment of Cloth in the Piece

In an initial series of experiments on the treatment of cloth directly, a commercial sample of olive-drab dyed herringbone twill was used. Samples were carboxymethylated as follows:

Approximately 8-x-8-inch pieces of cloth were soaked in a 40-percent aqueous solution of monochloroacetic acid containing about 0.1 percent of a wetting agent, the excess was squeezed out, and about 100 percent pickup of acid by weight of cloth was retained. They were then mercerized at room temperature, without tension, with 28- to 50-percent sodium hydroxide solution for 1 hour, and washed free of sodium hydroxide. One of these samples was lightly treated, another was more heavily treated, and the last was the heavily treated sample converted to the copper salt of carboxymethylcellulose. A mercerized control was prepared by soaking a piece of cloth in 40-percent sodium hydroxide solution for 1 hour.

This series of experiments resulted in a cloth product with greatly increased air permeability and decreased water permeability, as shown by the data in Table I.

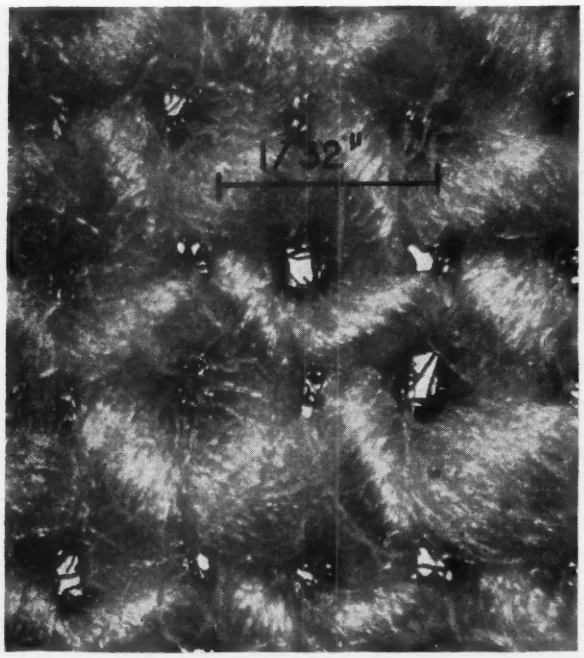
The treatment as applied to sample No. 3 increased the air permeability approximately four times over that of the control, decreased the water permeability by a factor of about 28, and affected hand and feel only very slightly. The heavier treatment given to sample No. 4 produced a rather stiff, harsh product. Conversion to the copper salt of carboxymethylcellulose (sample No. 5) resulted in very high air permeability, with at least some decrease in water permeability, which showed that the addition of copper did not entirely prevent swelling.

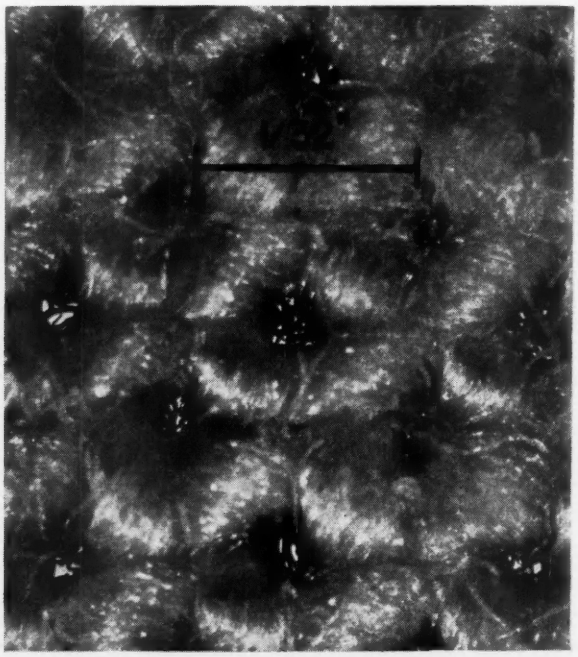
In order to show the swelling effect visually, one piece of loosely woven cloth was treated with 14-percent monochloroacetic acid and 35-percent sodium hydroxide to obtain approximately 1 carboxymethyl group per 20 glucose units. Another identical piece of cloth was mercerized with 35-percent sodium hydroxide to be used as a control. These two pieces of cloth were photomicrographed in the dry and wet states (Figures 1 and 2). The loosely woven material was selected to accentuate the interstices and the effect of swelling thereon. Figures 1B and 2B (mercerized and carboxymethylated cloth, respectively) illustrate the greater swelling capacity of the treated cloth. The white spots on the figures are holes. The dark spots are shadows in valleys due to the lighting arrangement used in photographing; light was passed through the cloth from below and crosswise from the sides.

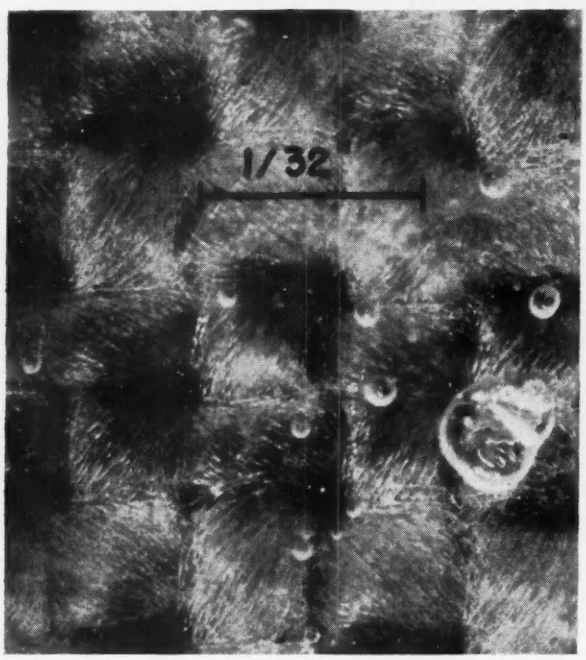
The two sets of samples described above had been treated without tension—that is, without regard for shrinkage resulting from the mercerization step. In another series of experiments the original dimensions

* Part I of this series appeared in the October, 1947, issue, page 554.

† One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.







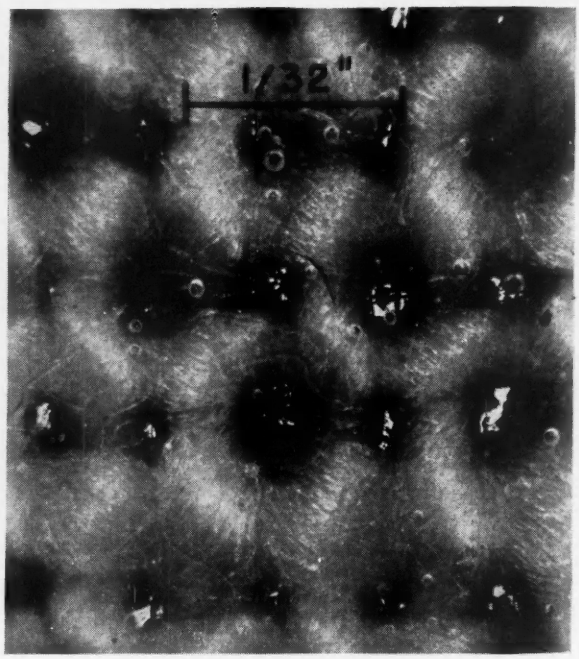


FIG. 1A (*top*). *Mercerized cloth* (*dry*).
FIG. 1B (*bottom*). *Mercerized cloth* (*wet*). *Same cloth as in Fig. 1A.*

FIG. 2A (*top*). *Carboxymethylated cloth* (*dry*).
FIG. 2B (*bottom*). *Carboxymethylated cloth* (*wet*). *Same cloth as in Fig. 2A.*

TABLE I. AIR AND WATER PERMEABILITIES OF CARBOXYMETHYLATED COTTON CLOTH MERCERIZED WITHOUT TENSION

Sample	Copper equivalent* (%)	Ratio of acid groups to glucose units	Air permeability† (cu.ft./min./sq.ft.)	Water permeability‡ (ml.)
1. Blank	0.0	—	16.8	10,430
2. Mercerized control	0.0	—	11.0	8,980
3. Treated	1.43	1:13	64.0	334
4. Treated	3.02	1:5	15.2**	3
5. Copper salt of sample No. 4	3.02	1:5	72.0	6,120

* Samples were converted to the copper salt of carboxymethylcellulose and copper analysis was run to determine degree of substitution [7].

† Determined on tester of National Bureau of Standards type [8].

‡ Determined on hydrostatic tester with 6-inch head for 5 minutes [3].

** Low result probably due to bonding of fibers on drying.

of the cloth were retained by carrying out the reaction under tension, as follows:

Cloth samples were soaked in various concentrations of monochloroacetic acid in aqueous solutions and the excess was squeezed out. They were then bolted between double stainless-steel frames (10 x 10 inches) and the dimensions thus obtained were maintained throughout the treatment. The framed cloth was immersed in 35-percent sodium hydroxide solution for 1 hour and then washed with distilled water until it was free of base. A set of samples was run using a tightly woven cloth (cotton drill 51 x 74). Results of the tests on this series are shown in Table II.

These data show that cloth treated under tension to the proper degree of substitution possessed greatly increased air permeability and somewhat decreased water permeability over the control samples. When the air permeability was increased too greatly—for example, by the heavier treatment (apparent in sample No. 14)—the larger interstices allowed the passage of more water and nullified the effect of the swelling of the yarns in the treated cloth.

A comparison of Tables I and II shows that tension has considerable effect upon the results, probably owing to increase in the size of the interstices.

Cloth from Carboxymethylated Yarn

In the preceding experiments it was shown that cotton cloth treated in the piece without tension had greater swelling capacity (Table I, samples Nos. 2 and 3), as well as greater air permeability, than the control. It seemed, therefore, that an advantage should be gained by treating yarn prior to its being woven into cloth since the interstices of the cloth would then be sufficiently small that the swellable yarns would be able to seal them on becoming wet. The experiments described below were conducted to test this premise.

TABLE II. AIR AND WATER PERMEABILITIES OF CARBOXYMETHYLATED COTTON DRILL CLOTH MERCERIZED UNDER TENSION

Sample	Monochloroacetic acid (%)	Sodium hydroxide (%)	Copper equivalent* (%)	Ratio of acid groups to glucose units	Air permeability† (cu.ft./min./sq.ft.)	Water permeability‡ (ml.)
6. Plain cloth	—	—	—	—	29.6	413
7. Mercerized control	—	35	—	—	88.5	432
8. Treated	4	35	1.17	1:20	136.0	289
9. Treated	12	35	1.35	1:15	95.5	350
10. Treated	16	35	2.08	1:8	211.5	440

* Samples were converted to the copper salt of carboxymethylcellulose and copper analysis was run to determine degree of substitution [7].

† Determined for 5 minutes on a hydrostatic leakage tester similar to the one described in reference [3] except that it had a 2-inch-diameter circle and a 25-cm. head.

‡ Determined on Gurley Permeometer with pressure drop equivalent to 0.50 inch of water.

TABLE III. AIR AND WATER PERMEABILITIES OF CLOTH WOVEN FROM MERCERIZED AND CARBOXYMETHYLATED YARNS

Sample No.	Weave	Description		Thread count		Weight (oz./sq.yd.)	Warp strength (lb.)	Air permeability* (cu.ft./min./sq.ft.)	Water permeability† (ml./5 min.)
		Warp	Filling	Warp	Filling				
11	Plain	Mercerized	Mercerized	62	48	7.55	104.9	14.8	169
12	Plain	Treated	Mercerized	62	48	7.99	101.0	18.0	106
13	Plain	Mercerized	Treated	61	48	7.54	104.1	16.4	103
14	Plain	Treated	Treated	62	48	7.91	100.6	19.5	113
15	Oxford	Mercerized	Mercerized	64	49	7.47	111.4	21.5	206
16	Oxford	Treated	Mercerized	64	50	8.02	114.3	25.3	174
17	Oxford	Mercerized	Treated	64	48	7.50	119.6	31.4	177
18	Oxford	Treated	Treated	64	48	7.74	122.3	33.3	192
19	Twill	Mercerized	Mercerized	62	48	6.70	120.7	62.0	265
20	Twill	Treated	Mercerized	61	50	7.53	127.2	67.0	167
21	Twill	Mercerized	Treated	62	48	7.37	117.9	72.0	209
22	Twill	Treated	Treated	62	48	7.49	113.2	76.0	118

NOTE: Results shown are average of ten determinations.
* Determined on Gurley Permeometer with pressure drop equivalent to 0.50 inch of water.
† Determined on Bundesmann machine [1] adjusted for 70 ml./min. and used without wiper blades.

Hand-Loom Woven Cloth

Skeins containing 400 yards of 3-ply 15s cotton yarn were soaked for 1 hour in 12-percent monochloroacetic acid in water containing a wetting agent, were padded twice, and squeezed free of excess solution. Previously a warp had been made of carboxymethylated singles yarn but weaving proved impractical because fuzz accumulated on the reed of the loom and caused breakage of warp threads. The skeins were placed on rollers, adjusted to a length of 27 inches, and mercerized with 38-percent sodium hydroxide solution. After standing for 30 minutes, they were washed with water and the remaining sodium hydroxide was neutralized with dilute acetic acid. The skeins were dried in a blower oven at 50°C.

A second portion of the 3-ply yarn was mercerized for control purposes. Four types of plain-weave cloth were woven from these yarns: cloth A, with mercerized warp and filling; cloth B, with mercerized warp and treated filling; cloth C, with treated warp and mercerized filling; and cloth D, with treated warp and treated filling.

The air permeability of sample A was 29.17 cu.ft./min./sq.ft.; sample B, 28.13; sample C, 35.10; and sample D, 35.90. The results again showed that carboxymethylation gave greater air permeability than could be obtained in untreated cloth.

Trial hydrostatic leakage tests on these samples showed that leakage was excessive and that the test was therefore not suited to this material. The Bundesmann test [1] was substituted but nevertheless the results were not reliable because individual samples of each type were nonuniform owing to the crude hand-woven character of the cloth. With the Bundesmann test, in 5 minutes sample A leaked 191 ml.; sample B leaked 120 ml.; sample C leaked 121 ml.; and sample D leaked 102 ml. This showed only that there was some swelling of treated yarn, which reduced leakage.

Power-Loom Woven Cloth

Because of the nonuniformity of the cloth woven on the hand loom, an attempt was made to overcome this difficulty by the use of a power loom. By means of false heads on the warp beam of a 42-inch loom, it was possible to use a 24-inch warp. About 12 pounds of cotton were needed for the experiment. This amount was spun into 2-ply 22.75s and the yarn was wound into skeins of 400 yards each.

In order to obtain uniform treatment, waxes were removed by extraction with hot alcohol in a large Soxhlet extractor. After the extraction, half of the yarn was mercerized to serve as a control and the other half was carboxymethylated to a substitution of approximately 1 carboxymethyl group per 20 glucose residues. The 24-inch warp was made up with 12 inches of mercerized yarn and 12 inches of treated yarn. The peg-pattern chain on the loom was set for a plain weave, and, with mercerized yarn used

as filling, 6 yards of cloth were woven; with treated yarn as filling, an additional 6 yards were woven. The peg-pattern chain was then changed for an Oxford weave and 12 yards of cloth were woven as before. In the same way, 12 yards of 45-degree twill were woven.

The power-loom weaving resulted in 12 different samples of cloth, 6 yards each, which could be compared not only for the effect of treated and untreated yarn but also for the three types of weave: plain, Oxford, and twill. Results are shown in Table III.

Although more uniform samples were obtained with the power loom, it is evident that the carboxymethylated yarns in the cloth did not swell enough to close the interstices sufficiently to prevent entirely the passage of water.

It is interesting to note that although the water permeabilities of the three weave types are approximately the same there are wide differences in the air permeabilities, the twill cloth having the greatest air permeability and the plain weave the least. The high rate of water absorption, to be described in the next section, offers an explanation of the rather disappointing resistance to water compared with the obviously large amount of swelling. Apparently the action of the sodium-substituted carboxymethyl group is similar to that of a wetting agent and thus allows the water to flow through the fabric easily, in a manner similar to that in which an organic liquid ordinarily flows through cloth. The experiment indicated that it was possible to weave carboxymethylated yarn on a power loom. The warp strengths shown indicate that there was very little, if any, degradation of the cotton by the treatment.

Dyeing and Absorption Characteristics of Carboxymethylated Cotton

The carboxymethylation of cotton alters its direct-dyeing characteristics. When treated cloth is dyed with a basic dye such as methylene blue, an intense blue color is obtained, whereas plain mercerized cotton is colored a very light blue. On the other hand, Solantine Blue, 4-GL (C. I. 922), a direct dye, imparts a rather dark blue to mercerized cotton but does not dye carboxymethylated cotton. These altered dyeing characteristics might be of interest for weaving in combination with untreated cotton when differential dyeing effects are desired.

It is also possible to form a salt of the carboxymethyl group with a diamine and, after diazotization, to couple it with a phenol or an amine. This reaction occurs with oxycellulose [6] because of the presence of carboxyl groups, but such material is ordinarily somewhat degraded. Some of the carboxymethylated cloth was treated with acetic acid to obtain the acid form and this was then allowed to react with benzidine in alcohol. Excess benzidine was removed by thorough washing with hot alcohol. The benzidine was then diazotized and coupled with phenols. Control samples were only lightly colored whereas the treated samples were dyed a deep orange when resorcinol was used and a deep red when β-naphthol was used as the coupling agent.

TABLE IV. ABSORPTION PROPERTIES OF CARBOXYMETHYLATED COTTON CLOTH

Sample	Substitution carboxymethyl/glucose	Absorption Static* gain (%)	Absorption Kettering method Time†	Absorption Kettering method Rate‡
23. Mercerized control	—	46.2	52	0.160
24. Treated	1:150	48.4	41	0.210
25. Treated	1:30	54.7	19	0.399
26. Treated	1:25	55.3	17	0.440

* Determined according to Standard Immersion Absorption Test [2].

† Seconds required for absorption of 0.2 ml. of water by approximately 2.5 g. of cloth by Kettering's method [5].

‡ Calculated by Kettering's method [5]:

$$\text{Absorption rate} = \frac{\text{Wt. water} \times 100}{\text{Seconds}} \div \text{Wt. of sample.}$$

Because of its obvious swellability, carboxymethylated cotton would be expected to have altered absorption characteristics. Studies were therefore made on samples with various degrees of carboxymethyl substitution for rate of absorption [5] and static absorption [2]. Results are shown in Table IV. The rate of take-up of water by treated material is approximately two to three times as great as that of the mercerized control, even though the total take-up is only slightly greater. This property might be of particular interest to the manufacturers of absorbent fabrics.

A comparison of the drying rate of treated and untreated cotton cloth was made as follows: Samples of treated and mercerized cloth containing approximately 40 percent of water were allowed to dry under standard conditions of temperature and humidity and both came to equilibrium or "dried" in about 3 hours, retaining approximately 8 percent of water. It was concluded that even though carboxy-

methylated cloth absorbed water faster than our mercerized control both fabrics dried at about the same rate.

Summary and Conclusions

The effect of the partial carboxymethylation of cotton has been studied on cloth treated in the piece and on cloth woven from treated yarn. The treatment of the cloth without tension yielded a product of satisfactory hand and feel with lower water permeability and greater air permeability than those of untreated material; cloth treated under tension had even greater air permeability but the water permeability was only slightly decreased.

Cloth was successfully woven from treated plied yarn but the expected decrease in water permeability was not attained. It should be possible, with slightly increased carboxymethyl substitution and a tighter weave, to obtain a cloth whose yarns would swell enough to close all interstices. Nevertheless, this effect would be somewhat counterbalanced by the tendency of the cloth to become completely wet, thus allowing water to penetrate the fabric more readily.

Treated cloth absorbs water two to three times as rapidly as untreated cloth although the total amount of water taken up is only slightly greater than that taken up by a mercerized control. The drying rate is about the same. The dyeing characteristics of carboxymethylated cotton are changed greatly.

The results indicate improvement of cotton for special uses where swellability or rapid absorption is desired. The addition of the carboxyl group may also serve as a starting point for additional chemical change in the characteristics of the cotton.

Acknowledgment

The authors are grateful to M. E. Jefferson for the photomicrographs; to Dr. Ruby K. Worner, Mrs. Leila D. Wiggins, and Miss Dorothy C. Legendre for various cloth tests; to Walter S. Lewis for some of the air-permeability results; to John J. Brown and L. Orr Bunton for spinning yarn and weaving cloth; and to Laurence W. Mazzeno, Jr., for aid in the treatment of yarn and in hand-loom weaving.

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(Manuscript received May 7, 1948)

A New Theory of Non-Linear Viscous Elasticity II: The Curve Shape Factor

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Contribution from the Textile Foundation, Frick Chemical Laboratory of Princeton University, and the Chemical Laboratories, Harvard University

IN A RECENT PAPER,† a new model for viscous elasticity, suggested by the $\alpha - \beta$ transformation in wool, was proposed. The treatment was based on the hypothesis that the molecules of the elastic material are capable of existing in two interconvertible configurations, a folded form of lower energy and a stretched form of higher energy. The theory of absolute reaction rates was applied to the model, yielding the differential equation governing the visco-elastic behavior.

In the present paper this equation will be reduced to one containing a single parameter, allowing a two-dimensional plot of the family of all possible stress-strain curves to be constructed. The shapes of the curves can then be investigated as a function of this parameter.

Following the notation of Paper I, it was shown that the two-state model was governed by equation (11) of that paper,

$$db/dt = (1 - b)DKe^{\alpha F} - bKe^{-\alpha F},$$

for a symmetrical energy barrier and neglecting molecular elasticity.

When solved using the auxiliary conditions of constant rate of elongation, constant rate of loading, or equilibrium (spring line) conditions, equation (11) gives a plot of F as a function of b in terms of the four parameters D, K, α, and the rate of elongation (or of loading). Because b is proportional to the elongation l, the resulting curves are the theoretical stress-strain curves for the material under consideration. The presence of four independent parameters, however, makes the problem of comparing these curves with experimental data a fairly difficult one; and it is therefore useful to change the variables to reduce the number of parameters in the fundamental equation.

Let

$$\alpha F = \psi - \ln \sqrt{D}. \tag{1}$$

Substituting equation (1) into equation (11) gives

$$db/dt = (1 - b)\sqrt{D}Ke^{\psi} - b\sqrt{D}Ke^{-\psi}$$

or

$$Gdb/dt = (1 - b)e^{\psi} - be^{-\psi}, \tag{2}$$

where

$$G = 1/\sqrt{D}K.$$

Equation (2) is now a two-parameter equation and its solution gives a plot of ψ as a function of b in terms of the two parameters. Since $\psi = \alpha F + \ln \sqrt{D}$, it is evident that the curve of ψ against b has the same shape as that of F against b, for the effect of the term $\ln \sqrt{D}$ is only to shift the zero of force, and the effect of α is to change the scale by a constant multiplicative factor.

Constant Rate of Elongation

A further reduction in the equation is now possible when the auxiliary conditions are taken into account. If constant rate of elongation is considered, $db/dt \left(= \frac{1}{l_x}\frac{dl}{dt} \right)$ is constant, and $Gdb/dt \equiv \beta = (1 - b)e^{\psi} - be^{-\psi}$.

Equation (2) is a quadratic in e^{ψ} and can be solved to give

$$\psi = \ln \left[\beta + \sqrt{\beta^2 + 4b(1 - b)}\right] - \ln 2(1 - b). \tag{3}$$

Using equation (3) one can plot the family of stress-strain curves. Because force, F, is a linear function of ψ (that is, $F = c_1\psi + c_2$, where c_1 and c_2 are constants) it is possible to compare the shapes of the force curves, divorced from their position or size, by adjusting the two parameters c_1 and c_2 so that the curves coincide as nearly as possible. This procedure is equivalent to giving arbitrary values to D and α, that are in general functions of β.

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** Mallinckrodt Chemical Laboratory, Harvard University, Cambridge, Mass.

† H. Burte and G. Halsey, TEXTILE RESEARCH JOURNAL **17**, 465 (1947). Hereinafter called "Paper I."

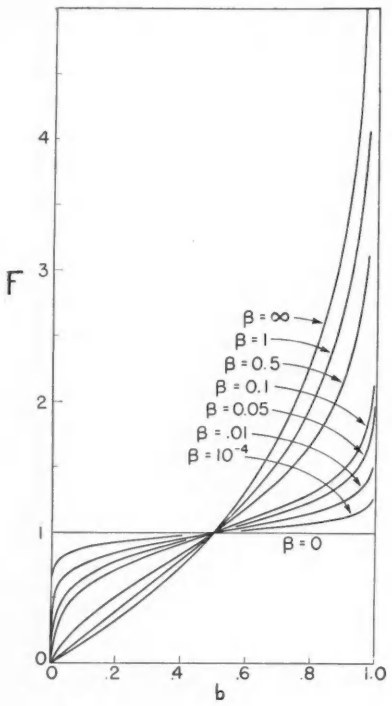

FIG. 1. *Stress-strain curves at a constant rate of elongation plotted according to case 1.*

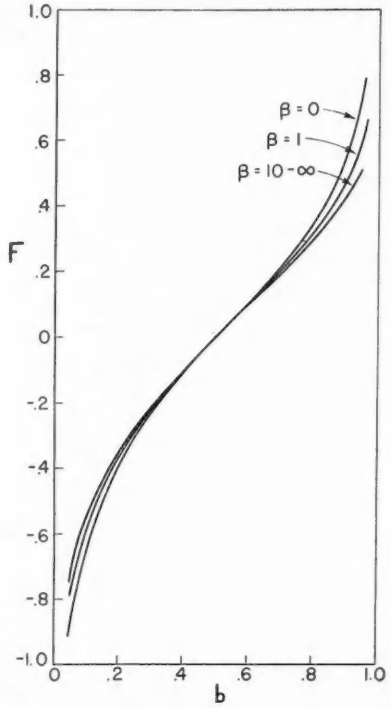

FIG. 2. *Stress-strain curves at a constant rate of elongation plotted according to case 2.*

Case 1—All curves pass through two common points.

In this case, all curves are to pass through the two points $F = 0$ at $b = 0$ and $F = 1$ at $b = \frac{1}{2}$, under which conditions

$$F = \frac{\ln [1 + \sqrt{1 + 4b\,(1-b)/\beta^2}] - \ln 2(1-b)}{\ln (1 + \sqrt{1 + 1/\beta^2})}. \quad (4)$$

Figure 1 is a plot of several representative curves at various values of β, including the two limiting cases: $\beta = 0 (F = 1)$ and $\beta = \infty$. For values of β greater than 25, the curves become almost coincident.

Case 2—All curves pass through one common point, with identical slopes at that point.

Here, the conditions imposed are that at $b = \frac{1}{2}$, $F = 0$, and $dF/db = 1$; from which one finds that

$$F = \frac{1}{2} \ln \left\{ \frac{1 + \sqrt{1 + 4b\,(1-b)/\beta^2}}{2(1-b)(1 + \sqrt{1 + 1/\beta^2})} \right\}. \quad (5)$$

Figure 2 shows the curves for various values of β. It is apparent that over the range $b = 0.2$ to 0.8 the curves lie close together for any value of the shape parameter. In practice, an attempt to reduce curves to coincidence using the procedure of case 1 would be very difficult, because it would imply knowledge of the value of F at $b = 0$, which is usually completely obscured owing to instantaneous elastic effects. The method of case 2 is easy to apply; both the position and the slope of the curve at its midpoint are most easily determined from experimental curves; these two data represent, in fact, the most accurately known portion of the curve. It thus becomes apparent that when the curves are adjusted to make this region coincide, the rest of each curve is close enough to coincidence with the others to make an accurate evaluation of the shape parameter impossible.

In order to evaluate the constants of the model, then, one cannot depend on shape, but must turn to the methods of comparing curves at differing rates, outlined in Paper I. It therefore emerges that a stress-strain curve can be represented, with reasonable accuracy, by using the spring-line curve, adjusted to pass, with the proper values of slope and position, through $b = \frac{1}{2}$.

Constant Rate of Loading

The solution of the constant-rate-of-loading problem cannot be carried out in closed form, but a

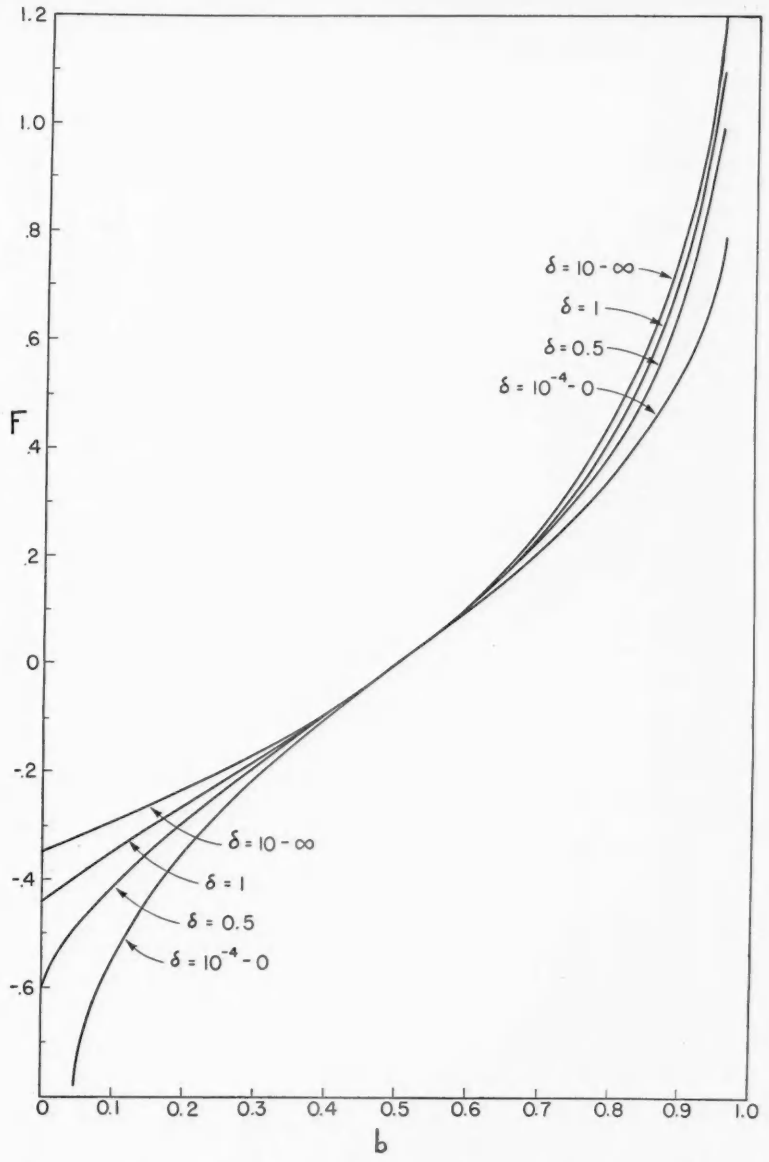

FIG. 3. *Stress-strain curves at a constant rate of loading, plotted according to case 2.*

numerical method can be employed to calculate the stress-strain curves. In Figure 3, the curves of F versus b so calculated are plotted for various values of the shape parameter $\delta = \alpha P/\sqrt{D}K$, where P is the (constant) rate of loading. The conditions of case 2 are employed. It appears that the conclusion reached concerning the constant-rate-of-elongation curves is not changed; the shape of the curves is here even more insensitive to changes of the shape parameter.

Summary

The four-parameter equation of Burte and Halsey (Paper I) has been transformed to a single-parameter equation. The solutions of this equation were investigated and, in each case, the shapes of the curves varied so little with changes in the parameter that the viscous constants could not be calculated accurately by comparison of these curves with those obtained experimentally. The application of this single-parameter equation, therefore, was found to be impracticable.

Acknowledgment

The authors wish to express their grateful appreciation to Dr. J. H. Dillon for his critical reading of the manuscript.

(Manuscript received July 2, 1948.)

Book Reviews

The Science of Plastics. Volume I. Edited by H. Mark and E. S. Proskauer. New York, Interscience Publishers, 1948. 632 pages. Price, $9.00.

(Reviewed by Lyman Fourt, Harris Research Laboratories)

This selected collection of the abstracts which have appeared in *Resins-Rubbers-Plastics Abstracts Service* in the five years 1942–1946 is not addressed primarily to textile workers, but will interest many in several ways.

Its special appeal is to those concerned with high-polymeric materials as the raw material of textile fibers, the chemistry of such materials, and the relation of molecular structure to mechanical properties. The presentation is scientific or fully technical, with emphasis on tests of theories and mathematical relationships. However, in the sections on elastic behavior, plasticity and flow, temperature effects, permeability and sorption, solvents and plasticizers, and laminates there is material to interest the practical user of plastics as coating materials for textiles or textiles as reinforcements for plastics. No material was observed, however, on resin finishing of textiles, which may reflect editorial concentration on basic theory and general properties.

Users of the abstract service will recognize that there has been extensive rearrangement and selection of the abstracts. The present volume contains general articles, or comparisons of numerous plastics; articles dealing with specific materials are reserved for Volume II. However, much information on specific materials can be located through the subject index. A rough count indicates that there are 228 abstracts, averaging over 2.5 pages each: The *Reader's Digest,* rather than *Chemical Abstracts,* is the model. Diagrams, tables, and figures from the original articles are reproduced. Although American and British papers predominate, German, French, and Russian papers are also represented.

The contents are indicated by the section headings: I. Properties and Evaluation—commentaries and general studies—tensile and impact strength—elastic behavior—plasticity and flow—wearing qualities—electrical properties—thermal expansion and related phenomena—permeability and sorption; II. Physical Chemistry of Polymer Systems—the solid state—solutions: fundamental theory, molecular weight, viscosity, osmotic pressure, light-scattering; III. Kinetics of Polymerization Reactions—mechanism and kinetics of vinyl polymerization—copolymerization theory—three-dimensional polymers and gelation; IV. Plastic Engineering—solvents and plasticizers—laminates—radio frequency heating.

Mechanical Behavior of High Polymers. Turner Alfrey, Jr. New York, Interscience Publishers, Inc., 1948. 571 pages. Price, $9.50.

(Reviewed by Robert Simha, National Bureau of Standards, Washington, D. C.)

This is the seventh volume to appear in the series on "High Polymers." The stated purpose is to present the "fundamental principles underlying the mechanical behavior of polymeric structures" and their relation to molecular characteristics. The general problem is, of course, a very difficult one, even in dealing with relatively simple systems of low molecular weight. Phenomena such as ultimate strength, fatigue, abrasion, and wear are but little understood from a theoretical point of view. On the other hand, visco-elastic behavior, crystallization, and related subjects can be approached theoretically by means of established methods; at least they can be described in terms of electrical or mechanical models. The book is concerned mainly with this second type of problem, on the basis of a unified approach which considers the various organic materials from which a fibrous, plastic, or rubber-like system is made up, as part of the general class of high polymers.

The phenomena treated are equilibrium (stress-strain-temperature effects) as well as rate processes (creep, relaxation, periodic stresses, and deformations). In the former, considerable attention is de-

voted to rubber-like elasticity. The experimental data selected for comparison with theoretical results are drawn from various types of organic polymers. The textile chemist and physicist will be particularly interested in the review of the crystallization and creep behavior of viscose, nylon, and wool. The discussion on a molecular basis is preceded by a description of the formal apparatus of elasticity theory, hydrodynamics, and their combination, useful in the analysis of visco-elastic behavior. In this connection the lack of suitable experimental data becomes apparent, when the often elaborate mathematical procedures are examined in relation to the meager results to which they can be applied.

It is to be expected that in a treatise of this kind the reader will not always find himself in agreement with the author's approach or interpretations. Readers of this JOURNAL might have desired a fuller consideration of non-Hookean and non-Newtonian behavior, within the limits of the period covered by this volume—that is, up to 1945.

The book will be valuable as a general survey to those working on other aspects of polymers. It will be of use also to those concerned with the mechanical behavior of any of the several types of polymers, in suggesting further experimental and theoretical research.

American Wool Hand Book. Second Enlarged Edition. Werner Von Bergen and Herbert Mauersberger. New York, Textile Book Publishers, Inc., 1948. 1,021 pages. Price, $8.00.

(Reviewed by F. W. Noechel, Botany Mills, Inc.)

The ten years that have elapsed since the printing of the first edition of the *American Wool Hand Book* have been fraught with wartime exigencies and manufacturing upheavals. The industry has progressed accordingly. The new book retains its valuable basic, fundamental material but it also takes into account the latest developments and processes. The book is divided into twenty-four chapters each one of which is a distinct account of the individual phases of the industry, from the historical and economical background to methods of physical and chemical testing.

The strategic value of wool during World War II and subsequent governmental controls are analyzed and considered as to their effect on the industry. Important changes in processes and equipment pertinent to mass production during the past 15 years are evaluated and explained. All aspects are well illustrated with tables, photomicrographs, charts, and pictures.

The chapter on physical properties of wool gives an enlightening picture of modern testing techniques such as x-ray, thermal transmission, stress-strain, moisture absorption and desorption. Tables illustrate the newest trends in research development. There is additional material on the chemical properties of wool, including a valuable account of the effects of alkalis, acids, and the like. Modified wool, nonfelting wool, and mothproofing are also discussed.

A report on new processes of worsted spinning, such as the American system, utilization of high drafts, and variable speed is presented in detail. An important table showing the twist constants for different types of yarns is of especial interest. The use of the Apperly Sliver Feed, Peralta Rolls, larger-package spinning frames, and other new equipment is described in the chapter dealing with woolen carding and spinning. The twist-tables given here will be useful to many manufacturers and will tend to standardize some phases of the industry. Warp preparation, design lay-outs, and weaving are fully covered and new equipment is described. The diagrams of loom-sections will be helpful in acquainting students with the intricate mechanisms of these machines.

Types of dyeing processes are illustrated, progressing from stock dyeing to top, yarn, and piece dyeing. The art of bleaching and printing and mothproofing processes are included in this section of the book. The chapter dealing with the classification of different kinds of finishes stresses the new streamlined machinery, with emphasis on reduction of manufacturing costs and uniform appearance of fabrics. The tables and diagrams illustrating rug and carpet manufacturing procedures give a clear picture of an industry which is an important part of the American way of life. Nonwoven and woven felt production is clearly outlined as to history and manufacturing methods. Numerous new testing devices and methods are described in the chapter entitled "Physical and Chemical Test Procedures." It is gratifying that the authors have included an account of statistical methods and the application of quality control. The omission of the section in the original edition dealing with garment dry-cleaning is regrettable inasmuch

as this industry has seen rapid growth. The many new-comers in this field need definite guidance.

There is a sizable bibliography of books, directories, catalogs, trade associations, and other publications. This section also contains a complete description of the Wool Products Labeling Act passed by Congress in 1940.

In presenting the second edition, the authors have brought their material up to date and have produced a well-rounded, basic "bible" for both the student and the wool man.

Research in Industry—Its Organization and Management. C. C. Furnas, Editor, New York, D. Van Nostrand Company, Inc., 1948. 574 pages. Price, $6.50.

(Reviewed by Lewis Sanders, Consulting Engineer)

This book is a philosophical and psychological discussion of the large industrial research laboratory, in twenty-nine chapters.

Each chapter is by a leading research man, or, in some cases, by two or more such men. In all, thirty-three men contributed, under the direction of an editorial board of seven. The editorial work has been well done; there is not much more overlapping of material in the various chapters than is frequently the case with a single author. The principal shortcoming of the book is its great length; its story could have been told far more effectively in half the space.

The book is concerned almost exclusively with the large industrial laboratory and emphasizes the philosophical and psychological factors influencing all phases of its operation, both internal and in its relations with the other departments within its own company and with the public.

The work is distinctly not a manual of laboratory operation and gives no direct answers to any questions. Its method is to resolve each phase of an industrial research laboratory's problems into its component parts, discuss each part separately, and then discuss its influence on the other phases of the problems. The reader is left to apply the same method of analysis to his own special case and to place his own relative value on each element of his own problem and thus to arrive at a sound decision. This is an eminently proper method and the one most likely to lead to good judgment, but one which will not satisfy the type of man who desires to find solutions to his problems all ready made because he wishes to avoid the labor of doing his own thinking.

The book should make an excellent textbook for such schools as the Harvard Graduate School of Business.

Action of Alkali upon Cellulose. Albert Schaeffer. Textile Series—Report No. 33. Washington, Hobart Publishing Co., Inc., 1948. 94 pages, 167 figures. Price, $5.00.

This is a translation of a report prepared in the laboratories of the I. G. Farbenindustrie by Dr. Albert Schaeffer, a well-known authority on cellulose.

It constitutes a systematic study aimed at increasing the durability of cellulosic fibers such as cotton and rayon. Data have been collected at different steps of the washing and laundering processes under various conditions to determine the action of the single components of the cleansing agents on cottons and rayons.

Chapter headings are: Investigation of the Effects of Common Laundering Agents; The Effect of Drying or Oxygen on the Alkali Boil; Chemical Reactions Possible During Treatment of Cellulose with Alkali in Presence of Oxygen; The Effect of Magnesium Silicate; Thermal Decomposition of Cellulose in the Course of the Drying Process.

Bibliography on High-Frequency Dielectric Heating. Ralph A. Rusca. New York, American Institute of Electrical Engineers, 1947. 19 pages. Price, $0.75.

This extensive bibliography of industrial applications covers technical publications from 1925 through July, 1946. It contains 393 literature and patent citations classified as "Domestic" and "Foreign."

ABSTRACTS

SERVICES AVAILABLE

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A list of periodicals abstracted, the abbreviations used in references to articles, and the addresses of publishers of English language publications is given in the January, 1948, issue, page 48.

ANALYSIS: TESTING: LABORATORY METHODS

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Ageing Cabinets

Construction and operation of accelerated ageing cabinets. D. S. Lyle. *Am. Dyestuff Reptr.* **36**, 623–8, 640 (Nov. 3, 1947).

The construction and standardization of ageing cabinets which may be used in conjunction with the Fadeometer is described. The cabinets provide a suitable means of controlling ageing conditions; they make it possible to expose fabrics to light in different atmospheres and to combine light treatment alternately with abrasion, laundering, and dry-cleaning treatments. The cabinets take sufficiently large specimens of fabric to permit numerous observations for strength, fluidity, and color change to be made.

Text. Research J. Sept. 1948 Author

Color Measurement

Apparatus for the color measurement of dyed textiles. P. M. Heertjes, E. van den Heuvel, and J. Lotichius. *Rec. trav. chim.* **66**, 749–57 (1947) (in English); cf. *C.A.* **42**, 1430*c* (through *Chem. Abstr.* **42**, 3181*e* (May 10, 1948)).

Text. Research J. Sept. 1948

Dielectric Measurements

Microwave dielectric measurements. T. W. Dakin and C. N. Works. *J. Applied Phys.* **18**, 789–96 (Sept. 1947).

The method of measurement of the dielectric properties of solid dielectrics described by S. Roberts and A. von Hippel has been applied with 3-cm. wave length waves in a rectangular wave guide. In this method the dielectric sample is placed at a short-circuited end of the transmission line, and the dielectric properties of the sample, calculated from the position of a minimum of the standing wave and the ratio of the minimum field strength to the maximum field strength of the standing wave. A simplified procedure for calculating the dielectric properties from the measurements is presented. Values are given of the dielectric properties of some common plastic and ceramic materials at 10-cm. wave length and 3-cm. wave length, which were described by this method. Authors

Text. Research J. Sept. 1948

Elastic and Plastic Deformation

The theory of combined plastic and elastic deformation with particular reference to a thick tube under internal pressure. R. Hill, E. H. Lee, and S. J. Tupper. *Proc. Roy. Soc.* (*London*) **A191**, 278–303 (Nov. 18, 1947).

In certain problems of plastic flow, *e.g.*, a thick tube expanded by internal pressure, it is important to consider changes in the elastic strain of material which is flowing plastically in order to deduce the correct stress distribution and deformation. The usual plastic theory which neglects elastic strains in the plastic region may lead to considerable errors in certain cases. The theory of the deformation of a material under combined stresses which involve both elastic and plastic components of strain is reviewed. The relationship between stress and strain is represented on a plane diagram, the reduced stress-strain diagram, which facilitates discrimination between the elastic and plastic components of strain and aids considerably the solution of certain problems. The diagram can also be used to express the relationships governing the dissipation of energy during plastic flow under combined stresses. The theory is applied to the deformation of a long thick tube under internal pressure with zero longitudinal extension. The solution is compared with that based on the usual theory which neglects elastic strains in the plastic region, revealing an error which reaches a maximum of more than 60% in the longitudinal stress

distribution. The significance of the differences between the two solutions is discussed. Authors
Text. Research J. Sept. 1948

Electron Microscopy

The use of diaphragms in the electron microscope. S. G. Ellis. *Can. J. Research* **25A**, 322–37 (Nov. 1947).

A method is described for deducing electron trajectories from optical measurements made on the electron microscope. A device for centering and changing apertures while the microscope is in operation is described. The choice of the size and position for diaphragms is discussed, together with other factors involved in their use. Author
Text. Research J. Sept. 1948

Electron Microscope Specimen Support

Films, resistant to organic solvents, for use in the electron microscope. S. G. Ellis. *J. Applied Phys.* **18**, 846–7 (Sept. 1947).

Alkathene films were prepared from hot xylene solutions which were suitable for specimen supports in the electron microscope. Soaps and carbons have no apparent solvent action on the films.
L. P. Witnauer
Text. Research J. Sept. 1948

Specimen Contamination in the Electron Microscope

Particle "growth" in the electron microscope. V. E. Cosslett. *J. Applied Phys.* **18**, 844–5 (Sept. 1947).

The author observed that particles of zinc, molybdenum, and magnesium oxide appear to increase in size when used as objects in the electron microscope. No increase in size was detected for gold-shadowed turnip virus particles or the flagella of bacteria. The effect is probably due to ejection of absorbed matter and perhaps the metal itself from the grid wires under bombardment.
L. P. Witnauer
Text. Research J. Sept. 1948

On the investigation of specimen contamination in the electron microscope. James Hillier. *J. Applied Phys.* **19**, 226–30 (Mar. 1948).

Experimental results and conclusions on the studies of the specimen contamination in the electron microscope for a great variety of substances are given. L. P. Witnauer
Text. Research J. Sept. 1948

Molecular Weight Determination

A demonstration of some new methods of determining molecular weights from the data of the ultracentrifuge. W. J. Archibald. *J. Phys. & Colloid Chem.* **51**, 1204–14 (Sept. 1947).

Methods of dealing with data obtained in an equilibrium-type ultracentrifuge, all of which are aimed at determining the molecular weight without waiting for equilibrium, are summarized. One method is based on the vanishing of a concentration function at the ends of the cell, and on the fact that whereas concentration at the ends is difficult to determine accurately and varies greatly with time, the function $r^{-1}n^{-1}dn/dr$, where n is concentration and r is radius, is constant at the ends. Curves of this function vs. r at various times come to common points at the ends of the cell; the value at these points is related to molecular weight. For a polydisperse solute the values at the two ends will differ and will vary with time, but if plotted separately vs. time will extrapolate to the same intercept at zero time, and this is the weight-average molecular weight. A second method depends on the demonstration that for a pair of values of r (which can be taken well away from the ends of the cell) such that a certain integral is zero, there is a function of $r^{-1}n^{-1}dr/dn$ which is related to the molecular weight. Many such pairs of values of r can be chosen for any one time of centrifuging, and the calculations repeated for other times, so that numerous checks on the value of molecular weight are obtained. Also, using the same data and curves, and selecting the two values of r so that the integral mentioned above is not zero, it is possible to determine values of the sedimentation and diffusion constants. Finally, a very simple method for determining a rough value of molecular weight is described, based on determination of the value of r at which the concentration remains approximately constant throughout the run. All the methods are illustrated by means of calculations based on an artificial set of data. W. E. Davis
Text. Research J. Sept. 1948

Molecular Weight of Polymers

Determination of the molecular weights of synthetic polymers. L. d'Or. *Rev. gén. caoutchouc* **24**, 425–31 (1947) (through *Chem. Abstr.* **42**, 2839*c* (May 10, 1948)).

A review and discussion of different methods for determining the molecular weights of polymers, with special attention to dynamic osmometry, the osmotic balance, light diffusion, and electron microscopy. 7 references.
Text. Research J. Sept. 1948

Viscosity Evaluation

The evaluation of the intrinsic viscosity (as intrinsic flow time) of GR-S in benzene. L. H. Cragg, T. M. Rogers, and D. A. Henderson. *Can. J. Research* **25B**, 333–50 (July 1947).

From careful measurements of the relative flow times of solutions of GR-S in benzene, it has been established that the intrinsic flow time (and hence the intrinsic viscosity) of GR-S in benzene may be most precisely determined by the use of a function $n(t_r^{1/n} - 1)/c$, based on the Baker equation relating η_r, the relative viscosity, and c, the concentration; for the GR-S-benzene system the value of n may be taken as 8. For the greatest precision $[t]$ is determined by linear (horizontal) extrapolation, to zero concentration, of the $8(t_r^{1/8} - 1)/c$ vs. c plot; in rapid routine work $[t]$ may be evaluated as $8(t_r^{1/8} - 1)/c$ by measurements on only one solution of a concentration such that $t_r = 1.8 \pm 0.4$. Authors
Text. Research J. Sept. 1948

A note on the variation with temperature of the intrinsic viscosity of GR-S in benzene. T. M. Rogers, D. A. Henderson, and L. H. Cragg. *Can. J. Research* **25B**, 351–6 (July 1947).

The intrinsic viscosity, $[\eta]$, (as well as the intrinsic flow time, $[t]$, and the intrinsic kinematic viscosity, $[\nu]$) of normal GR-S in benzene has been shown to be independent of temperature, over the range 10° to 55°C, within the experimental precision ($\pm\frac{1}{2}\%$). In this range of temperatures, also, the functions $8(t_r^{1/8} - 1)/c$ and $8(\nu_r^{1/8} - 1)/c$ are independent of concentration (up to at least 0.3%), and may therefore be used in the one-point determination of $[t]$ and $[\nu]$. Authors

Text. Research J. Sept. 1948

CHEMICAL AND PHYSICAL RESEARCH

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Adhesion to Cellulose

Theory of the adhesion of high polymers to cellulose. A. D. McLaren and C. H. Hofrichter, Jr. *Paper Trade J.* **125**, 96, 98–100 (Nov. 6, 1947); cf. *Ind. Eng. Chem.* **40**, 329–31 (Feb. 1948).

The adhesion of high polymers to cellulose involves a number of physical parameters including the dielectric constant of the polymer, the dipole moment of polymer constituents, the facilities for hydrogen bonding at the polymer cellulose interface and the micro-Brownian movement of chain segments. Here only the influence of polar group concentration and polymer viscosity have been considered. The concentration of carboxyl groups (maleic anhydride residues) was varied in a polymer composition of vinyl chloride-vinyl acetate copolymer admixed with vinyl chloride-vinyl acetate-maleic anhydride interpolymer. Such compositions were coated on regenerated cellulose. Experiments showed that adhesion to regenerated cellulose, which was assumed to be a measure of the COOH → OH dipole-dipole bonds formed, increases in a manner expressible by: Adhesion = k (COOH)^n, reminiscent of the Freundlich sorption isotherm. At any given carboxyl concentration adhesion increases with temperature. This may be interpreted as indicating that the development of an adhesive bond involves an exothermic heat of sorption as well as the highly endothermic activation energy for viscous flow. The latter energy is a measure of the process of the chain segment motion which is necessary to permit more dipoles within the polymer to approach the vicinity of the active centers in the substrate. Application of the Guzman equation relating the viscosity to the activation energy for viscous flow to the rheological measurements on the polymer composition shows this energy to be of the order of +30,000 calories (endothermic). Application of the Clausius-Clapeyron equation to the measurements of adhesion gives the heat of adhesion involved in the combined processes to be +12,500 calories. By difference, one may obtain the heat of sorption for maleic anhydride residues groups on cellulose to be approximately −17,500 calories (exothermic). Authors

Text. Research J. Sept. 1948

Direct Dyes on Cellulose

Physicochemical phenomena occurring in the dyeing of cellulose fibers with direct (cotton) dyes. Albert Schaeffer. *Textil-Praxis* **2**, 307–12 (1947) (through *Chem. Abstr.* **42**, 2775*b* (Apr. 20, 1948)).

The chemical structure and physical state of aggregation of a dye in solution is related to its permanent adsorption by cellulose fibers. It is proposed that adsorption of a direct dye by cellulose depends in general on the origin and nature of the fiber, the structure and chemical properties of the dye, the time, the dye concentration, and the concentration of electrolytes in the dye solution. Cellulose fibers can only adsorb dye molecules whose spatial dimensions correspond to intermicellar space in the fiber—i.e., the dye must be molecularly dispersed in solution before adsorption can take place. Direct (cotton) dyes are adsorbed by the fiber as neutral molecules, and once adsorbed they remain single molecules and do not form polymolecular aggregates on the fiber. The diffusion of the dye into the fiber and the subsequent adsorption are reversible processes which are related by dynamic equilibrium. Direct (cotton) dyes are long rodlike molecules, and the dye molecule is adsorbed in parallel with the cellulose micellar structure. In general, mercerized or other regenerated cellulose fibers adsorb more and better than cotton. Changes in the chemical structure of the cellulose fiber decrease dye adsorption.

Text. Research J. Sept. 1948

Hydrolysis of Wood Cellulose

Hydrolysis of wood cellulose and decomposition of sugar in dilute phosphoric acid. E. E. Harris and Bill G. Lang. *J. Phys. & Colloid Chem.* **51**, 1430–41 (Nov. 1947).

The data presented bear primarily on the problem of establishing optimum conditions for maximum yield of sugar in the saccharification of wood. However, the data on hydrolysis of Douglas fir wood and on decomposition of glucose, both in relation to time, temperature, and acid concentration (pH), are also of interest in connection with the problem of the relationship of hydrolysis rate to cellulose structure. W. E. Davis

Text. Research J. Sept. 1948

Light Diffraction

Diffraction of light by cellulose acetate yarn. E. V. Martin. TEXTILE RESEARCH JOURNAL **17**, 512–4 (Sept. 1947).

Filaments of cellulose acetate yarn, when properly prepared, have been found to have the property of diffracting visible radiation. Drafting the filaments at elevated temperatures under tension of about 25 mg. per denier produces in each filament 1 to 3 rows of regularly spaced cavities. These cavities are so regularly spaced along a line

parallel to the longitudinal axis of the filament that illumination of a filament, immersed in decahydronaphthalene, with parallel white light incident on the filament at right angles to the long axis will cause the filament to appear colored. The color of the fiber, when viewed at an angle θ to its longitudinal axis, varies according to the diffraction grating law $\eta\lambda = d \sin \theta$, where d is the distance between adjacent cavities. Since the relationship of cavity spacing to plastic draft factor is a linear one, this relationship can be employed to determine the approximate cavity spacing at the point of formation, and, since the cavities are not present in the undrafted fibers but are due to rupture in the early stages of drafting, extrapolation of the curve of spacing vs. draft factor to a draft factor of 1 gives an approximate value of the minimum spacing. This procedure yields a value of about 0.3 μ for the cavity spacing at the time of their appearance. The average length of the cellulose acetate molecules was calculated to be about 0.12 μ, indicating that the cavity spacing is not controlled primarily by the molecules themselves. The fact that these rows of regularly spaced cavities usually occur near a wrinkle in the outer wall of the filament would seem to indicate that the structure of the filament (possibly crystal structure) at these points plays a role in this phenomenon, although the mechanism by which it acts is not clear. Author

Text. Research J. Sept. 1948

Diffusion Theory

On a generalization in the diffusion theory. O. Lamm. *J. Phys. & Colloid Chem.* **51**, 1063–78 (Sept. 1947).

The scope and features of a theory of diffusion detailed in earlier works are outlined, including a simple proof of a generalized Sutherland-Einstein equation for the diffusion of a binary solution and the interpretation of diffusion measurements. A detailed treatment of the properties of the thermodynamic factor and the frictional coefficients involved, considering the chemical equilibrium between the components in arbitrary or dilute solutions, and the computation of an equation for the diffusion of electrolytes are given. A note is added on the diffusion of a ternary solution, this being most simply described in terms of volume fractions or moles per cc. Author

Text. Research J. Sept. 1948

Kjeldahl Nitrogen Determination

A critique of nitrogen determinations in amino acids and proteins by the Kjeldahl method. J. F. Reith and E. J. Wansink. *Chem. Weekblad* **43**, 803–9 (1947) (through *Chem. Abstr.* **42**, 2308*g* (Apr. 10, 1948)).

Sixteen current methods are critically discussed. Copper as catalyst gives low results; in some cases K_2SO_4 + Se gives good results, in others too low results; K_2SO_4 + HgO always gives good results but opinions differ on duration of heating. With respect to albuminous substances (casein) sulfate + Cu is held to be unsatisfactory. The combinations sulfate + Se, and sulfate + Cu + Se are praised by some, rejected by others; losses of N increase as the concentration of Se or sulfate increases; no objection has been raised to sulfate + Hg, which gives optimal results with long or short heating ($\frac{1}{2}$-5 hrs.). In experiments on histidine, oxyproline, tryptophan, lysine, and tyrosine the authors found that K_2SO_4 + Hg as catalyst yielded more N from all 5 amino acids than Se + $CuSO_4$ + Na_2SO_4 but the difference with tyrosine was small. Discontinuance of heating immediately after clearing gives too low results; the duration of heating should be determined for the apparatus used; $\frac{1}{2}$ hr. appears generally sufficient.

Text. Research J. Sept. 1948

Law of Particle Size Distribution

The mathematical description of certain breakage mechanisms leading to the logarithmico-normal distribution. B. Epstein. *J. Franklin Inst.* **244**, 471–7 (Dec. 1947).

It has been observed that the particle size distribution obtained from some breakage processes appears to be logarithmico-normal. There have been virtually no attempts to explain this phenomenon but in this paper a statistical model is constructed for a breakage mechanism which will generate size distributions which are asymptotically logarithmico-normal. In this model, $F_n(x)$, the distribution function of the particle sizes, after n steps of the breakage process, is the distribution function of the product of $(n + 1)$ independent random variables and hence, by the central limit theorem, is asymptotically logarithmico-normal. Author

Text. Research J. Sept. 1948

Permeability of High Polymers

The permeability of high polymers. J. R. Houwink. *Ind. plastiques* **3**, 409–14 (1947); cf. *C.A.* **41**, 7812*h*; TEXTILE RESEARCH JOURNAL **18**, 189 (Mar. 1948) (through *Chem. Abstr.* **42**, 2158*c* (Apr. 10, 1948)).

The mutual attraction between high polymers and pervious molecules explains the absorption of such molecules by high polymers. The permeability (P) in high polymers of gases or liquids depends on the solubility, h, and the coefficient of diffusion, D. Hence: $P = hD$. The solubility of polar gases in polar high polymers is greater than in nonpolar polymers; hence SO_2 and NH_3 are much more soluble in Perbunan (Buna N) than in polyisobutylene (Butyl). The diffusion in high polymers (rubber-type) of a gas is given by: $D = D_0 e^{-E/RT}$, where D_0 is a constant and E the energy of activation. It is shown that the energy of activation is a function of the molecular diameter of the gas. By using Henry's law it is shown that the variation in solubility of H_2O in rubber is a function of the partial pressure of the vapor, but the law does not hold above 75% partial pressure. The effect of plasticizer or softener

on the permeability of high polymers depends on their chemical nature. Not enough data are yet available to state a general rule.
Text. Research J. Sept. 1948

X-Ray Studies of High Polymers

Röntgenographic studies of synthetic high-molecular compounds. V. Crystal structure of synthetic nylon fibers. Keiroku Fuchino, Sampei Yasui, and Ichiro Sakurada. *Bull. Inst. Phys. Chem. Research* (Tokyo) **21**, 1055–63 (1942), *C.A.* **41**, 6104*d*. **VI. Change in the crystal structure of nylon at high temperature.** Keiroku Fuchino, Masaya Tanaka, and Sampei Yasui. *Ibid.* 1064–8 (through *Chem. Abstr.* **42**, 1779*i* (Mar. 20, 1948)).

When the temperature was raised gradually, no change in the x-ray pictures was noted in nylon bristle or fused mass below 150°C, or in nylon thread below 50°C. At 200°C. in the bristle and mass and at 100°C. in the thread *A3* and *A4* equatorial interference points fused into one; this indicates the coincidence of the 200 plane with the 400 plane, similar to the transition phenomenon reported by Fuller, *et al.* (*C.A.* **33**, 9083[1]) in paraffins below the melting point. This transition, perhaps caused by a vigorous torsional vibration of the long-chain molecules induced by the rising temperature, increased the crystal symmetry and formed a hexagonal cluster system, and mechanically decreased Young's modulus. After cooling, the material regained its normal crystalline structure. **VII. Crystal structure of polyamide from azelaic acid and hexamethylenediamine.** Keiroku Fuchino, Sampei Yasui, and Ichiro Sakurada. *Ibid.* 1069–76. Polyhexamethylenazeldiamide having an odd number of atoms in the chain, which was synthesized from azelaic acid and hexamethylenediamine, showed a fairly sharp x-ray picture in a fused mass but in the thread only after a heat-treatment below the melting point. The unit cell has $a = 7.8$ Å., $b = 40.15$ Å., $c = 5.3$ Å., and $\beta = 87°$. The fiber period is shorter than that of a planar zigzag chain, perhaps because the CONH linkage lies slightly out of the plane. Since the plane on which CO lies is at right angles to the main axis, the meridional reflection from the even-order position is strong. The chains are aligned in serration, the 1st and 3rd molecules being perfectly aligned but the 2nd being displaced laterally one-atom distance and so on. This polyamide showed a transition in the crystal structure similar to that in nylon as the temperature rose. **VIII. Heat-treatment of polyvinyl alcohol.** Ichiro Sakurada and Keiroku Fuchino. *Ibid.* 1077–83. When polyvinyl alcohol was once heated approximately at 200°C., it became more resistant to water, especially hot water, by an increase in the intermolecular tension due to dehydration by the rising temperature and the consequent constriction of the lattice space, which heightens the crystallizing tendency. A minute water content helped this tendency perhaps because some OH radicals in polyvinyl alcohol became free to form a new hydrous linkage. **IX. Röntgenographs of polystyrene and polyvinyl acetate at high temperatures.** Keiroku Fuchino, Masaya Tanaka, Sampei Yasui, and Ichiro Sakurada. *Ibid.* 1084–97. At room temperature both polystyrene and polyvinyl acetate gave 2 liquid interference rings, the inner ring being due to the intermolecular interference and the outer to the intramolecular interference. Both seemed to be composed of zigzag chains whose axes are arranged in a hexagonal cluster system. As the temperature was raised gradually to 200°C., the intramolecular interference became weaker and dispersed but the interval remained unchanged, while the intermolecular interference was not much weakened but the interval widened. The intermolecular interval reached a maximum above the softening point, widening at the rate of 1.2 Å. per 100°C., but after liquefaction widened further only slightly. The softening seems to be the transition from a form liquid to a typical liquid.
Text. Research J. Sept. 1948

BLEACHING: DYEING: FINISHING

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Wool Scouring

Nonionic detergents in the scouring of raw wool. H. C. Borghetty. *Am. Dyestuff Reptr.* **36**, 676–8 (Nov. 17, 1947).

Certain derivatives of ethylene oxide, particularly of the Igepal type, possess properties which make them ideal for raw wool scouring; their chief advantage over soaps for this purpose is that they have high cleansing efficiency in neutral baths. A typical mill scouring procedure using Igepal CA is described and a scouring chart is presented giving complete data on a practical mill run. K. S. Campbell
Text. Research J. Sept. 1948

Chemistry in Dyeing

The role of chemistry in the development of dyeing and bleaching. Sidney M. Edelstein. *J. Chem. Education* **25**, 144–9 (Mar. 1948).

Historical survey with bibliography.
Text. Research J. Sept. 1948

Coprantine Union Dyes

Coprantine union dyes. Th. Egger. *Ciba Rev.* **6**, 2285 (1947) (through *Chem. Abstr.* **42**, 3179*h* (May 10, 1948)).

By the use of Coprantine union dyes in neutral bath followed by aftertreatment in the same bath with Coprantine salt, goods can be dyed in solid yellow to gray, red, and pure Bordeaux shades of good fastness to light, washing, perspiration, hot-pressing, and water. These dyes are suitable for dyeing knitting yarns, uncarbonized rags, or pulled waste of pale color ground, and for the piece dyeings of good-quality materials. With certain shades, fastness can be improved by adding 2% Lyofix SB concentrate to the last water rinse after dyeing. Black can be obtained either with Union Fast Black DB or, where chrome

is not available, with Union Fast Black FO, which requires after-treatment with CH_2O and AcOH only.

Text. Research J. Sept. 1948

Effect of Electrical Charge

The effect of electrical charge in the application of dyestuffs. Rhode Island Section, AATCC. *Am. Dyestuff Reptr.* **36**, P697–702, P704 (Dec. 1, 1947).

There is no evidence that any of the direct and acid colors studied were affected by any electrophoresis (faster migration of dye ions toward the fibers of opposite charge) at the electrical potential applied in this work. The dyeing properties of many direct and acid colors can be materially affected by local changes in pH of the dye bath caused by electrolysis resulting from the application of electric current. In the range of current and potential studied, there is no evidence that the pickup of vat color pigments is appreciably affected by the action of electric current on the charged pigment particles. Some cationic dispersing agents materially increased the pickup of vat pigment particles, making practically complete exhaustion possible. By varying the pH of a vat pigment dispersion, the pickup can be controlled from a condition of practically no color pickup to one of almost complete exhaustion. Authors

Text. Research J. Sept. 1948

Fastness of Dyed or Printed Goods

Fastness to subsequent processing (of dyed or printed goods). Emil Krahenbuhl. *Ciba Rev.* **5**, 2191–2 (1947) (through *Chem. Abstr.* **42**, 3181*c* (May 10, 1948)).

Advantages of the treatment with Lyofix SB concentrate to improve the fastness of direct dyeings to subsequent processing in the production of colored discharges with vat dyes on direct dyed grounds are (1) improvement of the fastness to water, perspiration, and damp pressing, and (2) increased resistance of the dyed grounds to oxidizing agents used in warm developing baths in which untreated grounds are often stripped. The protective action is diminished considerably by prolonged steaming. Immediately after padding, the shade of many dyeings undergoes considerable change and the material acquires a somewhat harsh handle; usually these changes in appearance and handle disappear completely on steaming and rinsing.

Text. Research J. Sept. 1948

Sizing Problems

Sizing problems of the weaver and finisher. Karl Walter. *Monatschr. Textil-Ind.* **57**, Fachheft **2**, 50–319 (42) (through *Chem. Abstr.* **42**, 2439*a* (Apr. 10, 1948)).

The traditionally opposed aims of sizing processes in the weaving industry on one hand and in the finishing industry on the other hand are discussed. Modern simplified sizing compositions to be used by the weaver and their subsequent processing by the finisher are described. For water suspensions or solutions the following sizing baths are recommended: (1) For cotton yarns (*a*) potato starch 5–8, Aktivin S (a starch degrading agent) 0.05–0.07, and Olgon (a softening agent) 0.2% by weight; (*b*) Hortol S 25 2–2.5 and sizing wax 0.2%. (2) For cell-wool yarns (*a*) potato starch 3, Silkovan K powder 1, and Olgon 0.2%; (*b*) Tylose MGC 25 2 and Plexi glue 2%. (3) For mixed cotton cell-wool yarns, potato starch 4 and Nekal B Extra 0.1%. (4) For worsted yearns, potato starch 4 and Plexi glue 4%. (5) For woolen or mixed woolen yarns, Plexi glue 6–7%. (6) For viscose yarns, Silkovan K powder 1.5–2.5%. (7) For rayon yarns, Blufajo 4–5%. (8) For acetate yarns, Silkovan K powder 8%. (9) For linen yarns, potato starch 2–4 and Plexi glue 2–4% by weight. To identify these different types of sizes the following reactions are indicated: for starch-containing sizes: I_2 in KI; for albumin-containing sizes: the biuret or ninhydrin reaction. The finisher can remove these sizes easily by treatment of the goods with diastatic desizing agents or water. German wartime regulations of the trade are discussed briefly.

Text. Research J. Sept. 1948

Treatments for Cotton Duck

Weather protective treatments for cotton duck. Southeastern Section, AATCC. *Am. Dyestuff Reptr.* **36**, P705–10 (Dec. 1, 1947).

Data are presented which show that among various experimental finishes investigated urea-formaldehyde used alone has definite weather protective properties for cotton and is even more effective when combined with pigments. Certain pigments applied *in situ* and pigments bound with oil-modified alkyd resin also effected a high degree of weather resistance. Exposure to sunlight in a normal-incidence cabinet while isolated from other factors of weather was rated in the same order of effectiveness as outdoor exposure. A comparison of results of outdoor exposures with those of carbon-arc exposures did not reveal a definite correlation, but indicated that the weathering machine can aid in classifying the ultraviolet screening properties of experimental treatments. Authors

Text. Research J. Sept. 1948

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

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Rayon Staple

Rayon staple technology—The key to the future in textiles. Harold Ashton. *Can. Text. J.* **65**, 40, 42–3 (Apr. 30, 1948).

Outstanding problems to be solved if the textile industry is to command the full potential of staple fibers are: (1) the development of a satisfactory sliver-making technique over the full range of desirable fiber dimensions, and (2) the development of long drafts with control over the full range of desirable fiber dimensions. Desirable fiber dimensions include a free range over staple lengths from 1 to 4 in., with a filament denier range from 1 to 8 to be

used in any combination. The merits of "Rayolanda" as a complementary fiber for blending with either wool or cotton are discussed. The possibilities of spun-dyed colors in this field of staple fiber are also traced. L. A. Fiori

Text. Research J. Sept. 1948

Spinning Rayon

Cotton system easily adapted to rayon staple. Anon. *Textile World* **98**, 141, 230, 232, 234 (Feb. 1948).

The cotton system is adaptable to the spinning of rayon staple without major changes. The adjustments of the machines used for each stage of processing are described.

A. L. Landau

Text. Research J. Sept. 1948

Mule Spinning

Production in mule spinning. R. Jackson. *Textile Weekly* **41**, 296, 298 (Feb. 13, 1948).

This article indicates some of the factors, e.g., room temperature, lineshaft drives, rimshaft speeds, and drawing-in speeds, which contribute to the efficient running of the mule spinning machine. For maximum production and, at the same time, peak efficiency, it is necessary to experiment with pulley diameters on the lineshaft, countershaft, and rimshaft until an acceptable combination is attained. Examples illustrating the reasons for this experimentation are given.

Text. Research J. Sept. 1948 L. A. Fiori

Ring Spinning

The movable spindle rail. M. Sattanathan. *Textile Weekly* **41**, 540, 542, 544 (Mar. 19, 1948).

A description of the movable spindle rail ring spinning frame. Advantages claimed are increased efficiency, higher production per man hour, and fewer end breaks. With the new arrangement, it is possible to produce soft yarns with comparatively lower twist multipliers and with as high a breaking strength as yarns which are produced on the mule spinner. Another advantage is the possibility of producing larger packages than is customary with ordinary frames; this is achieved without imposing extra strain on the yarn. L. A. Fiori

Text. Research J. Sept. 1948

Fabric Shrinkage

Shrinkage of certain cotton woven fabrics caused by various laundering and pressing procedures. H. M. Fletcher and M. V. Jones. *Am. Dyestuff Reptr.* **36**, 727–9 (Dec. 15, 1947).

Results show that mechanical action affects the shrinkage of woven cotton fabrics. In home laundering the shrinkage was consistently less than when the fabrics were subjected to mechanical action both in the wash wheel and in the tumbler during drying, irrespective of the method of pressing. Maximum shrinkage, which is not produced until after the third laundering, can be measured by the standard test method if it is repeated successively for at least 3 times. Multiple tests of 4 or 5 launderings would be adequate for predicting shrinkage in cotton woven goods. The method of pressing affects the amount of shrinkage. A procedure in which tension is exerted along the warp and filling of the fabric results in considerably less shrinkage than a procedure in which no tension is exerted. Authors

Text. Research J. Sept. 1948

Rebeaming Cotton

Cotton rebeaming. Anon. *Text. Mercury and Argus* **118**, 210 (Feb. 6, 1948).

A rebeaming machine is described comprising 2 three-step pulleys driven by V-belts and a ball-bearing jockey pulley with a quick release handle to relieve tension. Three winding speeds on a warp beam weighing 400 lbs. can be attained.

Text. Research J. Sept. 1948 L. A. Fiori

Reeding

Applying reeds with a minimum of error. H. E. Wenrich. *Rayon Text. Mo.* **29**, 62–4 (Mar. 1948).

The fundamentals of applying a reed—reed condition, preparation for cross in harness, stitching the reed, checking for errors, and preparing the completed job for storage or transportation—are described in detail. Methods for checking the accuracy and correcting errors of reeding and practical hints for facilitating the reeding operation are also given. L. A. Fiori

Text. Research J. Sept. 1948

Selvage Imperfections

Selvage imperfections. G. Creasey. *Textile Age* **2**, 66–70 (Feb. 1948).

A discussion of the causes and remedies of selvage defects caused by weaving with over-tensioned filling yarns. (*Cf.* TEXTILE RESEARCH JOURNAL **18**, 197 (Mar. 1948).) A. L. Landau

Text. Research J. Sept. 1948

Synthetic Proteins

Know your synthetic protein fibers. G. H. Brother. *Plastics* (*Chicago*) **7**, 52, 54–6 (Oct. 1947).

Describes the development of synthetic protein fibers, contrasts their properties with those of the native fibrous proteins, and discusses the reasons for the differences. The principal drawback in using fibers such as casein is the low tensile strength, particularly when wet, and this is attributed to the lack of primary valence cross-links. Methods whereby such cross-links might be supplied are discussed, and a new method of preparing casein fibers of higher orientation (and therefore greater dry tensile strength) is described.

W. E. Davis

Text. Research J. Sept. 1948

Woolen Carding

Modern methods of woolen carding. R. W. Wright and A. Brearly. *Textile Recorder* **65**, 36–9, 59 (Dec. 1947); **65**, 46–8 (Jan. 1948).

Mechanical details of a new woolen card are described. An important prerequisite for this type of card is the use of neat oils in preference to most emulsions, and for normal blends about 10% of a good-quality oleine uniformly applied is advocated. With high speeds, excessive

lubrication may cause "rolling" of the material on the card. A variable-speed synchronized motor drive is featured which by means of a simple press-button control produces a speed range between 75 to 150 r.p.m. for the cylinders. It also allows individual parts to be put out of action as desired or reversed for grinding. A special production drive has been included so that the production of the entire set can be varied at will with a minimum amount of alteration. Card clothing, its method of application, suitable clothing gage or count for a wide range of blends, and methods and frequency of grinding and stripping, and particulars of operational speeds, production, and types of blends are given. Some advantages are (1) increased production, in some cases, doubled; (2) the possibility of processing a wide range of qualities and counts on the same machine; and (3) production of superior yarns free from neps.

Text. Research J. Sept. 1948 L. A. Fiori

Increasing the efficiency of the woolen carding department. P. P. Townend. *Textile Recorder* **66**, 49–51 (June 1948).

Factors affecting the efficiency of woolen carding are described and suggestions for improvement are given. L. A. Fiori

Text. Research J. Sept. 1948

Worsted Carding

Worsted carding. Anon. *Platts Bulletin* **6**, 9–12 (Jan.–Feb. 1948).

Constructional and operational differences between the two systems of carding—the woolen and the worsted—are described and illustrated. Speeds, clothing counts, and general make-up of the 2 carding machines are analyzed. Appended is a table of card-clothing counts and roller speeds for 64s quality Merino combing wools.

Text. Research J. Sept. 1948 L. A. Fiori

Worsted Drawing

Modern trends in worsted drawing. Anon. *Can. Text. J.* **65**, 43–4 (Apr. 2, 1948).

Research in worsted yarn production has been devoted mainly to increasing speed and hence increasing production or eliminating processes in order to reduce costs and labor. Attention is being directed to possible methods of reducing open drawing costs by modifying the number of processes in a set of drawings. A reliable method of gaging yarn quality with respect to yarn variation and color blending is to knit a length of it and compare it with the knitted web. Short-term variations are produced mainly in the late stages of drawing and in spinning; long-term variations, in the early drawing processes. An attempt is made to overcome them by "balancing" at the can gill box and at the first drawing. If these early stages are omitted, long-term variation diminishes. Both skein and single-strand strength tests on yarn produced from 5 drawing operations against 9 show no loss in strength. L. A. Fiori

Text. Research J. Sept. 1948

MISCELLANEOUS

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Cotton Textile Industry

Cotton cloth. War changes in industry series no. 27. U.S. Tariff Commission. Washington, U.S. Government Printing Office, 1947. 142 pages. 40 cents.

Output of cotton cloth in United States increased from 9,000,000,000 sq. yds. per yr. during 1937 and 1939 to over 12,000,000,000 sq. yds. in 1942. Since 1939 there has been little new investment and only a limited quantity of new machinery with increased production due to longer hours of operation. World trade in cotton cloth was declining for a number of years before World War II owing to growth of local industries in former importing countries. Exports of cotton cloth are vital to such countries as United Kingdom, Japan, Italy, Belgium and it is expected that these countries will endeavor to recover former markets. The tariff problem is discussed. R. B. Evans

Text. Research J. Sept. 1948

Latin American Fiber Industry

Status of the fiber plant industry in Latin America. Brittain B. Robinson and Tallmadge Bergen. Washington, D.C., Pan American Union, December, 1947. 170 pages.

Status of each Latin American country with respect to production, requirements, imports, exports, and local manufacture of vegetable fibers other than cotton, including jute, flax, hemp, henequen, sisal, abaca, phormium, caroa, guaxima, and others, is discussed. Latin America as a whole produces a surplus of hard fibers and still has vast undeveloped resources, but is extremely deficient in soft fiber production. It has been shown by actual production that hard fibers such as sisal, henequen, fique, cabuya, letona, cocuiza, etc. can be produced, manufactured, and used for domestic requirements for sacks and cordage in many Latin American countries. This production is beyond the early experimental basis both in agriculture and manufacture. With increasing population, a growing merchant marine, and the opening of new areas, Latin American demands for cordage, twine, and bagging can be expected to increase. It is recommended that present fiber industries should maintain their production for the next 2 to 5 yrs. because of uncertain economic and political world conditions. R. B. Evans

Text. Research J. Sept. 1948

Australian Flax Industry

Flax in Australia. Anon. *J. Text. Inst.* **39**, P116 (Mar. 1948).

A brief historical review of the Australian flax industry, from its inception to the present.

Text. Research J. Sept. 1948 L. A. Fiori

Indian Jute Industry

Future prospects of Indian jute industry. P. C. Jain. *Indian Text. J.* **48**, 217–19 (Dec. 1947).

The Indian jute industry produced to excess capacity between 1929 and 1939 and was further stimulated

by World War II but coal, labor, transport shortages, and the requisition of mills by the government reduced and dislocated the output. Prices were decontrolled in Oct. 1946 after about 2½ yrs. of price control and by Aug. 1947 the price of raw jute had increased 67%, the price of jute bags had increased 70%, and the price of jute cloth had increased 132%. These prices are three to four times higher than prewar prices and they may encourage production of jute in other countries and development of substitutes. The jute mills located in the Indian Union fear that their supply of jute, mostly from Pakistan, may be upset and are therefore anxious to encourage increased production in their own country. Output in this area has been declining because of diversion of acreage in raising food crops but could be greatly expanded by use of better seed and more manure. Reduction in the number of jute mills in India is advocated. Cost of producing jute goods can be lowered by using better cultivation methods, which can double the present yield of less than 3 bales per acre, and by increasing efficiency of the processing industry.

Text. Research J. Sept. 1948 R. B. Evans

Nylon in Industry

New industrial application for nylon. Anon. *Textile Recorder* **66**, 51–2 (May 1948).

The use of nylon in tire cords, filter cloths, laundry nets, anode bags, etc., in the various industries is outlined. L. A. Fiori

Text. Research J. Sept. 1948

Health Hazards in Rayon Industry

The artificial fibers industry and its dangers. J. Auffret. *Arch. maladies profess., méd. travail et sécurité sociale* **7**, 181–96 (1946); *Chimie & industrie* **57**, 573–4 (1947) (through *Chem. Abstr.* **42**, 2436*f* (Apr. 10, 1948)).

A discussion of the health hazards of the viscose industry and of medical and technical measures of overcoming them.

Text. Research J. Sept. 1948

Parkinson's Disease from Carbon Disulfide

Parkinsonian trembling in a workman exposed to carbon disulfide vapors. H. Desoille, M. Gaultier, Goulene, and Assouly. *Arch. maladies profess., méd. travail et sécurité sociale* **7**, 140–2 (1946); *Chimie & industrie* **57**, 575 (1947) (through *Chem. Abstr.* **42**, 2439*h* (Apr. 10, 1948)).

A workman exposed for 18 yrs. to CS_2 vapors in a rayon-spinning mill exhibited marked static trembling of the upper and lower left limbs, such as is observed in Parkinson's disease. In CS_2 parkinsonism there are generally character changes and motor deficiencies; in the present case there were no character troubles, sexual functions were normal, and the peripheral nerves undamaged, but chronaxic examination revealed chronaxia changes similar to those observed in sulfocarbonism. In spite of a lack of absolute certainty, the trembling would seem to be attributable to CS_2.

Text. Research J. Sept. 1948

Jacquard Loom

Fine-pitch centre-shed Jacquard. Anon. *Textile Weekly* **41**, 1028, 1030 (May 28, 1948).

The latest model Dracup centre-shed Jacquard loom incorporates 3 striking features: (1) an endless paper card instead of thick durable card with one for each pick, (2) no contact between card and the ordinary needles, and (3) slotted castings by means of which the shed can be quickly changed from straight lifts to tilted shedding. Illustrations are given. L. A. Fiori

Text. Research J. Sept. 1948

Developments in Machinery

Developments in textile machinery and appliances. Joseph S. Barke. *Indian Text. J.* **58**, 424–6 (Feb. 1948).

A new cloth-printing machine, a method of removing waste threads on a woolen card, a self-aligning shuttle-box device, and a single new building motion for speed frames are reviewed briefly. L. A. Fiori

Text. Research J. Sept. 1948

Mixing and Blending

Mixing and blending. R. Z. Walker. *Textile Bulletin* **74**, 51, 52, 54 (Feb. 1948).

Modern opening room procedures are described.

Text. Research J. Sept. 1948

Punched Card Techniques

The use of punched card techniques in the coding of inorganic compounds. John C. Bailar, Jr., Karl F. Heumann, and Edwin J. Seiferle. *J. Chem. Education* **25**, 142–3, 176 (Mar. 1948).

Since the end of the war, the National Research Council, through its Subcommittee on Codification of Chemical Compounds of the Chemical - Biological Coordination Center, has continued work toward developing a classification system designed to include all chemical compounds. This article is a progress report of the Inorganic Panel of the Subcommittee and gives the rules so far developed for codification, along with an invitation for suggestions and criticisms.

R. K. Worner

Text. Research J. Sept. 1948

Reproduction of Reports

Reproduction techniques for reports and information service. B. H. Weil and John C. Lane. *J. Chem. Education* **25**, 134–41 (Mar. 1948).

A survey of methods of reproduction for use in presenting factual material in the most effective manner that is consistent with the economics of the situation. Typing, hectographing, stenciling, offset printing, multilithing, photo-offsetting and printing from type techniques, as well as reproduction of illustrations by photographic copying, photostating, and blueprinting are considered. Methods for binding loose sheets, card files, and abstract bulletins are also discussed.

R. K. Worner

Text. Research J. Sept. 1948

Slasher Combs

Slashers and slashing. A. E. Silcox. *Textile Age* **12**, 75, 76, 78, 80 (June 1948); TEXTILE RESEARCH JOURNAL **18**, 321 (May 1948); *ibid.* 445 (July 1948).

A description of the types of combs used on the head-end of slashers. Spring combs, positive expansion combs, and inclined dent combs are explained. A. L. Landau

Text. Research J. Sept. 1948

Slasher Cylinders

Slashers and slashing. A. E. Silcox. *Textile Age* **12**, 66–70 (Apr. 1948).

The slasher cylinder is described, detailing design of inner construction, safety valves, vacuum valves, and coverings. Stainless-steel covering is preferred to copper for some types of work. The number of cylinders per slasher is dependent upon the weight of the warp and the necessary drying rate.

Text. Research J. Sept. 1948 A. L. Landau

Slasher Lubricator

Slasher lubricator reduces warp and filling stops. Anon. *Textile Inds.* **112**, 92–5 (Mar. 1948).

Fats or lubricants which are commonly mixed with the size mix and applied to the yarn at the size box of the slasher are applied separately by means of a slasher attachment fixed between the size box and the cylinder. Application of the lubricant at this point, and separately from the size compound, has resulted in the reduction of both warp and filling loom stops.

Text. Research J. Sept. 1948 A. L. Landau

Synthesis of Starch

Enzymic synthesis of starch. Shlomo Hestrin. *Brewers Digest* **23**, No. 1, 43–6 (1T-4T) 50 (8T) (1948) (through *Chem. Abstr.* **42**, 2296*a* (Apr. 10, 1948)).

A review with 74 references.

Text. Research J. Sept. 1948

Weaving Machine

Warner & Swasey introduces Sulzer weaving machine. W. A. Newell. *Textile World* **98**, 110–13, 228, 230, 232 (June 1948).

A well-illustrated description of the new Warner & Swasey weaving machine which is claimed to operate at speeds in excess of 210 picks per min. Filling bobbins are completely eliminated as filling yarn from a cone or cheese is picked up by a shuttle and carried across the shed in one direction only.

A. L. Landau

Text. Research J. Sept. 1948

Woolen Machinery

Woolen machinery. Anon. *Fibres* **9**, 227–8 (June 1948).

A condensation of the English, Scotch, and American carding systems summarized in 4 sections: scouring and blending, carding machinery, leather tape condensers, and techniques. L. A. Fiori

Text. Research J. Sept. 1948

Wool Wax

Wool wax. D. T. C. Gillespie. *J. Text. Inst.* **39**, P45–85 (Feb. 1948).

The properties, recovery, and utilization of the actual fatty secretion in the fleece of the sheep are reviewed at length. Information is also given on the distribution of the wool wax in the fleece, the composition of wool wax, the products of wool wax, and the uses of its derivatives. A table of analytical and physical data is presented for the comparison of wool wax with beeswax, carnauba wax, lard, and cottonseed oil. 422 classified references. J. A. Woodruff

Text. Research J. Sept. 1948

Use of Wool Wax Acids

Some uses of wool wax acids. E. S. Lower. *Ind. Chemist* **24**, No. 276, 46–9 (1948) (through *Chem. Abstr.* **42** 2454*f* (Apr. 10 1948)).

Industrial utilization of wool wax acids (*I*) is suggested; standard chemical techniques are employed. Solvent-soluble carboxylic acid amides (*II*) are prepared by heating wool fat with ethanolamine for 10 hrs. at 170°C. *II* can be made to react with ethylene oxide and aqueous alkali to give a H_2O-dispersible product. Sulfonated derivatives of *I* are prepared by heating the sulfonated acids with amines. The resulting sulfonated amides can be made to react with HCHO at 130°–70°C to form waxy products. *I* is made to react with naphthylamine at 200°C to form arylides suitable for polishes. Ketones suitable for wax substitutes, dielectrical materials, plasticizers, and waterproofing agents can be prepared from *I*. Dialkyl ketones are prepared either by pyrolysis of Ca salts of *I*, or by catalytic reaction of the acids in the vapor phase. Aryl-alkyl products are prepared by reaction of the acid chlorides. Acid chlorides of *I* are prepared by reaction with thionyl chlorides, PCl_3, or PCl_5, and are useful mainly as intermediates. Amines of *I* are prepared by hydrogenation of the acid nitriles, and are useful, as are their salts, as surface-active agents. Chlorination of *I* in conjunction with the oxidation products of montan wax in AcOH solution gives a powerful emulsifier for bitumen, asphalt, or pitch. H_2O-absorbing preparations are formed by agitation with bone charcoal. Glyceryl esters are prepared by reaction with glycerol above 100°C, and are good emulsifiers for H_2O-oil systems. P compounds are prepared by reaction with P_2O_5. *I* can be compounded with rubber in normal tread compositions, replacing stearic acid and pine tar. S derivatives are prepared by treating with sulfonating agents in the presence of phenol. They are useful as dyeing assistants, in leather tanning, and as emulsifiers. Cf. *C.A.* **41**, 3308*h*, 7059*f*; **42**, 1074*g*.

Text. Research J. Sept. 1948

Wool Research

Research in wool textiles. *Textile Weekly* **41**, 940, 942, 944 (May 14, 1948); *Text. Mercury and Argus* **118**, 714–6 (May 14, 1948).

A report covering a comprehensive survey of the research work done by the Wool Industries Research Association. (Cf. *Textile Recorder* **66**, 54–7 (June 1948). L. A. Fiori

Text. Research J. Sept. 1948